Characteristics of a dynamic atomic force microscopy based on a higher-order resonant silicon cantilever and experiments

Huang Qiangxian a,⇑, Zhao Yang a,b, Yuan Dan a, Zhang Liansheng a, Cheng Zhenying a

a School of Instrument Science and Opto-Electronic Engineering, Hefei University of Technology, Hefei 230009, China
b College of Electronic and Information Engineering, Anhui JIANZHU University, Hefei 230601, China

A R T I C L E   I N F O

Article history:
Received 29 March 2014
Received in revised form 23 July 2016
Accepted 26 July 2016
Available online 27 July 2016

Keywords:
Sensitivity
Scanning speed
Higher-order resonance
Cantilever
Dynamic AFM
Surface scanning

A B S T R A C T

In order to improve the sensitivity and scanning speed of the dynamic AFM, a surface scanning method using higher-order resonant cantilever is adopted and investigated based on the higher-order resonance characteristics of the silicon cantilever, and the theoretical analysis and experimental verification on the higher-order resonance characteristics of the corresponding dynamic AFM cantilever are given. In this method, the cantilever is excited to oscillate near to its higher-order resonant frequency which is several times higher than that of the fundamental mode. Then the characteristic changes a lot compared with the first-order resonant cantilever. Because of the changes of the quality factor, amplitude and the mode shape of the cantilever, the higher-order resonant AFM gets higher sensitivity and scanning speed. Based on the home-built tapping-mode AFM experiment system, the resolution and the response time of the first and second order resonance measured by experiment are respectively: 0.83 nm, 0.42 nm; 1265 l/s, 573 l/s. The higher-order resonance cantilever has higher sensitivity and the dynamic measurement performance of the cantilever is significantly improved from the experimental results. This can be a useful method to develop AFM with high speed and high sensitivity. Besides above, the surface profile of a grating sample and its three-dimensional topography are obtained by the higher-order resonant mode AFM.

0. Introduction

A micro silicon cantilever is used in the atomic force microscope (AFM) to sense the atomic force between the tip on the cantilever and atoms on the sample surface, and then to realize surface scanning by the detected force signals. AFM is one of the instruments with highest spatial resolution by now. It has a sub-nanometer or even higher spatial resolution, which has been widely used in various field [1–4], such as nano-measurement, nano-manipulation, nano-machining and nano-etching [5–8].

The AFM is divided into two categories according to the operation mode of the cantilever: static mode AFM and dynamic mode AFM [9]. Nowadays, many versions of dynamic AFM have been proposed and developed [10–12]. All of these versions rely on the cantilever’s dynamic characteristic that the resonant parameters (amplitude or frequency/phase) of the cantilever are sensitive to the atomic interactions to make the measurement. Compared with the static mode AFM, the dynamic mode AFM has become the main working mode of the AFM because of its small lateral force, fast scanning speed, strong anti-interference ability and other advantages. In fact, the cantilever used in AFM has many resonant modes. The minimum detectable force gradient and dynamic response characteristics of these AFMs are restricted by the geometric parameters of the cantilever itself and cannot be further improved.

Currently, efforts are still done to make the dynamic AFM more sensitive and more repaid. It is believed that increasing resonant frequency is good to lower the minimum detectable force gradient and improve the scanning speed of the cantilever in dynamic AFM [13]. In order to obtain a higher operating frequency, fabrication of nanometric-scale cantilevers of small mass has been already reported [14]. Due to the small size of the cantilever, this kind of cantilever which requires more complicated detection systems, should be prepared specially and is difficult to be commercialized. Another alternative is to use conventional AFM silicon cantilevers vibrating at higher-order resonant modes. In earlier years, Rabe and Minne had done some research on the higher-order resonant mode AFM, and gave detailed analysis of cantilever vibration mechanisms [15,16]. Girard P concluded that the stabilization time of the second resonant mode cantilever is smaller than that of the first resonant mode cantilever in vacuum and ambient conditions.
Recently, the research on the higher-order resonance has gone further with some new theories applied to the vibrating model analysis of cantilever [18], and multifrequency AFM [19–21] and subsurface AFM emerged [22,23].

Since the commercial silicon cantilever has many resonant modes and the cantilever in higher-order resonant mode is more sensitive to the micro external force, a scanning method based on the higher-order resonance mode of the cantilever is proposed. The higher-order resonant characteristics of the silicon cantilever will be analyzed and validated both theoretically and experimentally, and so does the feasibility and superiority of the profile measuring by using this method.

1. The flexural vibration model of the cantilever

In dynamic AFM here, the cantilever is assumed to be a uniform, homogenous beam with constant, rectangular cross section. One end of the cantilever is fixed and the other is hung up for free vibrating, and a tip with a small radius (the mass of the tip is assumed to be zero) is attached to the free end. The flexural vibration schematic diagram of the resonant cantilever is shown in Fig. 1. Assuming that the deflection of any arbitrary point in the cantilever relative to its equilibrium position is defined by \( y(x,t) \), \( x \) is the horizontal distance between the point observed and fixed end of the cantilever, \( t \) is the time, the Young modulus, the inertia moment of the cantilever are defined by \( E \) and \( I \) respectively, \( EI \) is uniform over the length of the cantilever. \( L \) is the length of the cantilever. When the dynamic AFM's cantilever vibrates in atmosphere environment, the air damping exists inevitably and must be considered. Damping is modeled by parameter \( c \) which describes the additional damping coefficient caused by air when the cantilever vibrates and \( m \) is the mass density of the material. \( f(x,t) \) is the interaction force and it is the tip-sample interaction force at the point \( x = L \). Then the equation of motion for transversal cantilever vibrations is:

\[
EI \frac{\partial^4 y(x, t)}{\partial x^4} + m \frac{\partial^2 y(x, t)}{\partial t^2} + cm \frac{\partial y(x, t)}{\partial t} = f(x, t)
\] (1)

The transformation rule is \( y(x, t) = \sum_{i=0}^{\infty} \phi_i(x)Y_i(t) \). Here, \( \phi_i(t) \) and \( Y_i(t) \) are defined as the eigenfunction and the corresponding generalized coordinates of the \( i \)-th order resonant cantilever. After been integrated in the range \([0–1]\) and been applied the orthogonal properties of the vibration mode, Eq. (1) can be transformed into a set of decoupled ordinary differential equations in generalized coordinates:

\[
Y_i''(t) + 2\gamma_i \omega_i Y_i'(t) + \omega_i^2 Y_i(t) = \frac{F_i(t)}{M_i}
\] (2)

where the apostrophes denote the corresponding derivatives with respect to time \( \partial/\partial t \). In Eq. (2), the generalized mass of the \( i \)-th order resonant cantilever is \( M_i = \int_0^1 m(x)\phi_i^2(x)dx \), the generalized force of the \( i \)-th order resonant cantilever is \( F_i(t) = \int_0^L f(x,t)\phi_i(x)dx \). \( Y_i(t) \) is defined as the corresponding generalized coordinates. \( \omega_i \) is the resonant frequency and the damping ratio of the \( i \)-th order resonant cantilever. The form of formula (2) is same as that of standard single-degree-of-freedom systems, so the frequency response characteristics of high-order resonant cantilever can be solved as the form of Lorentzian curve [24]:

\[
A = \frac{A_{m}(\omega/\omega_1)}{1 + Q_i^2(\omega/\omega_1 - \omega_1/\omega_2)^2}
\] (3)

\( A_{m}, Q, \omega_1 \) is defined as the vibration amplitude, quality factor and resonant angular frequency of the \( i \)-th order resonant respectively.

The change of the tip-sample interaction forces drives shift of the vibration amplitude and the resonant angular frequency of cantilever. In this paper, the detection of changes in the tip-sample interaction forces was achieved by detecting the deformation in the vibration amplitude of the cantilever. Clearly, in order to get the biggest change in cantilever vibration for a given change in resonant angular frequency, one would work on the steepest portion of the \( A \) vs \( \omega \). This is not at the peak point but occurs at \( \omega_m = \omega_1(1 \pm 1/\sqrt{8Q_i}) \). At this point on the curve, the maximum slope of the \( A \) vs \( \omega \) given by

\[
\frac{dA}{d\omega} = \frac{4A_{m}Q_i}{3\sqrt{3Q_i}}
\] (4)

Two main factors that affect the amplitude deformation of resonant cantilever are vibration amplitude and quality factor of the cantilever. The experimental results show that the \( Q \) of the second order is much higher than the first order under the same experimental conditions. These are favorable factors for improving the detection sensitivity.

In a dynamic AFM system in which the vibrating amplitude of the cantilever is detected by optical lever method, the deflection angle of the cantilever can produce a corresponding optical power change \( \Delta P(x, t) \) on position sensitive detector (PSD) [25] as

\[
\Delta P(x, t) = \sqrt{2}\pi p_d \frac{P_{d0}}{\lambda} \theta(x, t)
\] (5)

where \( p \) defines the incident optical power, \( \lambda \) is the wavelength of laser and \( d_0 \) defines the beam diameter projected on the cantilever. The power change \( \Delta P(x, t) \) on PSD is proportional to the deflection angle \( \theta \). The deflection angle in the free end of the first, second, and third order resonant cantilevers are \( \theta_1, \theta_2, \theta_3 \) respectively. It can be seen from Fig. 3 that the deflection angle in the free end of the

![Fig. 1. Schematic view of a rectangular cantilever at the higher-order resonant frequency interacting with sample.](image1)

![Fig. 2. Curve of amplitude-frequency characteristic.](image2)
higher order resonant cantilever is obviously larger than the first order when they have same amplitude. The amplitude variation sensitivity of the higher order resonant cantilever detected by PSD is expected to be higher than the first order, which can also be seen from the following experimental results of the approach curves and the test of the height of steps.

Therefore, because of the changes of the quality factor, amplitude and the mode shape of the cantilever, the higher-order resonant AFM gets higher sensitivity compared with the basic mode AFM system.

Besides the higher sensitivity and resolution, the cantilever in higher order resonant mode has good dynamic response. In dynamic AFM, the response time of the cantilever is proportional to expression $2Q_i\omega_i^{-1}$ [14]. As the increasing of the resonant frequency, the response time of the cantilever decreases. Therefore, the scanning speed of the dynamic AFM has the potential to be improved if the cantilever is in higher-order resonant mode.

2. Experimental system

The main hardware structures of the higher order resonant AFM consist of two parts: the AFM head and the AFM stage. The AFM head includes the fixed frame, the bending deflection detection unit for silicon cantilever, the semiconductor laser, and the adjusting mechanism for laser light path, etc.; the AFM stage includes the three dimensional piezoelectric scanner, the sample stage, and the rough adjustment, approaching system between the tip and samples.

The scanner used in this system is a three dimensional piezoelectric ceramic tube (PZT), and the micro displacement in x, y, z directions is achieved simultaneously under the feedback control by electronic control system. The functional block diagram of the AFM system is shown in Fig. 4. The bending vibration of the cantilever is detected by optical lever method. The vibration signals of the cantilever are received by four quadrant position sensitive detector, and are sampled by A/D Converter after the processing of pre-amplification, sum and differential operation. Then the difference between the acquired data and the preset voltage is sent to a high-voltage amplifier and drive the scanner and sample up and down by keeping the consistent amplitude of the cantilever while moving the scanner along X and Y directions smoothly. The surface topography of the sample can be achieved by the real-time recording and reconstruction of the scanner location by data processing.

This AFM is a multi-mode system. It can operate in contact mode, the first order resonant dynamic mode, the second order resonant dynamic mode and even higher-order dynamic mode.

3. Experimental results and analysis

For AFM, the sensitivity of the cantilever, the vertical spatial resolution in z direction, and the dynamic response of the AFM are important parameters for scanning performance.

In experiment, a kind of ContAl tip from Budget Sensor was chosen, with its force constant 0.2 N/m. Fig. 5 shows the frequency spectra of the first, second, and third order and the enlarged view of each resonance peak. The resonant frequencies of the first, second, and third order are 15850 Hz, 97100 Hz, 277800 Hz respectively, with their ratio 1:6.13:17.53, which are coincide with the calculated results [11]. The quality factors of the first, second and third order resonant cantilever are 63, 176, 168, respectively. In a bandwidth of 1 Hz, the vibration amplitudes of the first and second order were measured with their values about 140 nm, 40 nm respectively.
According to the quality factors and resonant frequencies of the first, second, third cantilever, and the formula $s = \frac{2}{Q_i}$, where $s$ defines the response time of the cantilever, the response time obtained by test for the first three order resonant cantilevers are 1265 $\mu$s, 573 $\mu$s, 191 $\mu$s respectively. From the obtained response time, higher-order resonant cantilevers have small response time and quick mechanical response. This means that the AFM scanning speed can be improved as the cantilever operates in a higher-order resonant mode.

According to the Eq. (5), the amplitude variation of the second mode resonant cantilever is larger than the first order under the same force gradient. That is to say, the sensitivity of the amplitude variation of the second mode is larger than the first order. The approach curves shown in Figs. 6 and 7 are obtained by the first and second order resonant cantilever by detecting the resonant amplitude signals of the cantilever using the optical lever method.

It can be calculated from Figs. 6 and 7 that the slope of the approach curve obtained by the first order resonant cantilever is 6 V/µm, while 12 V/µm for the second order resonant cantilever. It is verified experimentally that the sensitivity of the amplitude variation of the second mode is larger than the first order. In the experiments, the resonant signal noise of the cantilever is about 5 mV, which determines the spatial resolution in vertical direction along with the sensitivity of the cantilever. The experimental data also verify that the spatial resolution of the second order resonant cantilever is higher than the first order with their resolutions 0.42 nm, 0.83 nm respectively obtained from the approach curves. This result is in coincidence with the theoretical conclusion that the sensitivity of amplitude displacement of the higher order resonance is smaller than the first order resonance.

Furthermore, resolution test of AFM in different resonant modes was carried out. Ten continuous time-equidistant steps in Z direction with heights from 1 nm, 2 nm, ..., 10 nm are generated by Z PZT stage. The distance between the tip and the sample changes correspondingly and the responses of the cantilever to different steps are detected. The minimum step that the cantilever can detect is the resolution, and this is the most intuitive way to describe the resolution of the cantilever. Figs. 8 and 9 show the schematic view of the steps and the cantilever responses to different step heights.

It can be seen from Fig. 9 that the first step with the height of 1 nm can be distinguished clearly, but from Fig. 8 the first step cannot be distinguished obviously. In order to test the resolution of the first and second resonant cantilever furthermore, ten steps in Z
direction with heights from 0.1 nm to 1 nm was measured and Figs. 10 and 11 show the cantilever responses to different step heights. It can be seen from Fig. 11 that the fifth step with the height of 0.5 nm can be distinguished, while from Fig. 10 the ninth step with the height of 0.9 nm can be detected. The results show clearly the second order resonant cantilever is more sensitive than the fundamental mode with their vertical resolution 0.5 nm and 0.9 nm respectively.

In order to verify the scanning validity of the higher order resonant cantilever, a grating with its nominal pitch 1.12 µm and height 330 nm was scanned by the home-made AFM mentioned above. In this experiment, the cantilever was driven to vibrate in its second order resonant mode, and the amplitude feedback control of the AFM in vertical direction was adopted. Fig. 12 shows the three-dimensional topography of the grating obtained by the second order resonant cantilever, with its scanning range 3 µm in X direction, step 20 nm, and 0.2 µm in Y direction, step 40 nm. Fig. 13 shows one of the line profiles of the grating. It can be seen from the scanning results shown in Figs. 12 and 13 that the grating pitch is 1.2 µm and height is 340 nm which is approximate to its nominal value, while the slight difference in pitch, height and profile is resulted from the nonlinearity, creep and three dimensional non-orthogonal movement of the ceramic tube in working stage [26]. Even though, the experimental results have proved the feasibility and effectiveness of the AFM system based on the second order resonant cantilever, and the surface profile scanning by higher order resonant cantilever is realized.

4. Conclusions

A surface scanning method based on the higher-order resonant characteristic of the AFM cantilever was adopted, and an AFM in which the cantilever operated in higher-order resonant mode was developed. The superiorities of the higher order resonant cantilever in sensitivity, spatial resolution and the minimum detectable force gradient were proved theoretically and experimentally by using the home-made AFM system compared with that of the first resonant cantilever. This method provides more probabilities to develop dynamic AFM with high speed and high sensitivity. The experimental results were as follows:

(1) The existence of the higher-order resonance phenomenon was verified through the frequency spectra curve, with the resonant frequency ratio consistent with the theoretical results, and it’s also concluded that the higher-order resonant cantilever had higher spatial resolution, and faster scanning speed.

(2) The sensitivity and spatial resolution of the second mode were higher than the first mode, with their sensitivity 12 V/µm, 6 V/µm respectively obtained from approach curves, and spatial resolution 0.42 nm, 0.83 nm respectively determined by signal noise and sensitivity. On the other hand, this conclusion is verified through the steps test, with their resolution 0.5 nm, 0.9 nm respectively.

(3) The surface of a silicon grating sample was scanned and its three-dimensional profile was obtained by the second order resonant cantilever. The feasibility of using higher order resonant cantilever was proved.

Obviously, the higher-order approach has the advantages of higher sensitivity and shorter response time. However, there are some disadvantages that need to be concerned further. Compared to the traditional AFM, the cantilever oscillation of the higher order is a bit more difficult. It is found that the resonant amplitude at the second order is usually smaller than that of the fundamental mode.
when the cantilever is excited with the same voltage in the experiments. Besides, when the cantilever is driven to vibrate in the second order mode, there is a point in the cantilever where the angular deflection is zero. If the laser focused spot is located at this point, the sensitivity is zero. The distance between the point and the free end is about 0.58 L. In order to get better sensitivity, the position that the laser beam can be projected on should be far away from that point and as close as possible to the free end of the cantilever. Therefore, the adjustment of optical lever system is more strictly required at higher-order mode. If those disadvantages are avoided, the higher-order approach could be used in the future.

**Acknowledgments**

This work was supported by a grant from National Natural Science Foundation of China (Project No. 5147531), Natural Science Foundation of Anhui Provincial Department of Education (Project No. KJ2015JD09) and the Fundamental Research Funds for the Central Universities (Project No. JZ2015HGQC0212).

**References**