SIMPLE EVALUATION OF ELASTICITY IN NANO-METER SCALE USING A FLAT-ENDED SENSOR TIP IN SENSITIVITY-ENHANCED ATOMIC FORCE ACOUSTIC MICROSCOPY

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ABSTRACT

Atomic force acoustic microscopy (AFAM) producing high-resolution images reflecting sample elasticity is a powerful tool to observe nano-structured materials. Quantitative evaluation of elasticity in AFAM, however, is difficult and challenging because of the following two problems. Resonant frequencies, as a measure of sample elasticity, of a micro-cantilever equipped with a sensor tip deviate from the classical beam theory especially for higher modes. In addition, modeling the contact stiffness between a tip and a sample is complicated by uncertainties in the tip shape and presence of the adhesion force. Our previous work has introduced a mass-concentrated (MC) cantilever to enhance sensitivity in detecting elasticity for stiff samples. MC cantilevers also eliminate the former problem since the vibration obeys well the one-freedom theory (i.e., spring-mass model). This study proposes a flat-ended tip as a unique shape of sensor tips so as to delete the latter problem. A flat-ended tip keeps a constant contact area. Therefore the contact stiffness becomes independent of the adhesion force as well as of the contact force. A MC cantilever with a flat-ended tip significantly simplifies evaluation of sample elasticity. A Ti/Pt-coated silicon tip with a radius of 35nm was plastically deformed to make the tip flat-ended. Etched Si(100) wafer and diamond were selected for standard samples having known elastic coefficients. Together with use of a theoretical relation of the resonant frequency vs. the contact stiffness, measurements of resonant frequencies for these samples determined reasonable values for the radius of the contact area (4.4nm) and the effective Young's modulus of the tip (152GPa). Etched Si(111) wafer was evaluated for its effective Young's modulus by use of the theoretical relation, the resonant frequency measured for the sample and the pre-determined values of the radius and the tip elasticity. It confirmed that the present evaluation agrees with the effective Young's modulus of bulk sample with an error of about one percent.

1 INTRODUCTION

Mechanical reliability designs of microstructures like micro-devices and micro-mechanical-electronic systems frequently require mechanical properties such as elastic modulus for thin films and for extremely narrow areas in the nanometer range. Near-field acoustic microscopy such as micro-deformation microscopy (Cretin and Sthal [1]) and atomic force acoustic microscopy (AFAM) (Rabe, et al [2]) is a promising technique of evaluating such local elasticity. In AFAM, the resonant frequency of a micro-cantilever equipped with a sensor tip gives a measure of the contact stiffness between a sample and a tip (see Figure 1).

For stiff samples like metal and ceramics, AFAM is confronted with significantly low sensitivity, i.e., the resonant frequency insensitive to the contact stiffness. The vibration modes of higher orders and the stiffer cantilevers improve the sensitivity (Rabe, et al [3]). They also impose difficulty in measuring high frequencies over mega hertz and low sensitivity in controlling the contact force, which leads to large contact forces resulting in low spacial resolutions. A mass-concentrated (MC) cantilever proposed by Muraoka [4] gives an ideal solution for the problem. MC cantilevers always produce the maximum sensitivity in detecting elasticity for any sample even for stiff materials without any additional difficulty and sacrifice. Figure 2 shows an example of AFAM images by using a MC cantilever. The elasticity image clearly shows the heterogeneity of a grain boundary in a titanium film sample. It also reveals dark (i.e., stiffer) patterns inside the grain, which seem dislocation piling like slip bands,

Quantitative evaluation of elasticity is challenging in AFAM. It requires both of a vibration model relating the resonant frequency to the contact stiffness and a contact model relating the contact stiffness to the elastic modulus of sample. The classical beam theory has been adopted as the vibration model but explains not well measurements of contact resonant frequencies especially at higher orders of mode. In addition, an appropriate contact model is complicated due to not simple shapes of real tip (not spherical tip) and presence of the adhesion force. Use of MC cantilevers for enhancing the sensitivity can also eliminate the former problem. MC cantilevers are designed so as to obey well the elemental mass-spring model and provide a simple relationship between the resonant frequency and the contact stiffness.

This study proposes a flat-ended tip as a new type of sensor tip to eliminate the difficulties in modeling the contact stiffness. A flat-ended tip maintains a constant contact area independent of the adhesion force as well as of the contact force. It drastically simplifies a relationship between the contact stiffness and the elastic modulus of sample. Evaluation of sample elasticity by a combination of a MC cantilever and a flat-ended tip is demonstrated for standard samples with known values of elastic coefficients: diamond, etched Si(100) wafer and etched Si(111) wafer. Resonant frequencies measured for diamond and Si(100) are analyzed inversely to determine a radius of the contact area and an effective Young's modulus of the tip. Resonant frequency measured for Si(111) is utilized to assay precision in the present evaluation of sample elasticity.

2 EXPERIMENTAL PROCEDURE

2.1 A mass-concentrated cantilevers with a flat-ended tip

The mass-concentrated (MC) cantilever was constituted of a rectangular cantilever made of single-crystalline silicon [NSC12, Type F, MikroMasch; (thickness 2μm)×(width 35μm)×(length 250μm), spring constant k_c =1N/m, fundamental resonant frequency 47.2kHz] and a tungsten (W) particle as a concentrated mass. When determining the mass m_c of the particle, it was noticed that

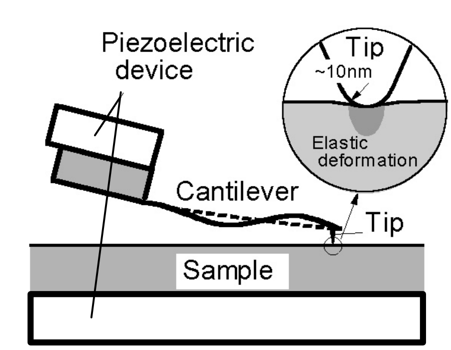
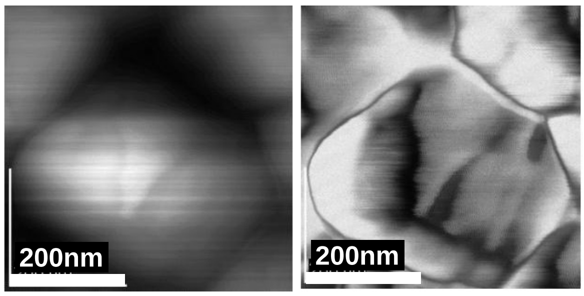


Figure 1: Principle of detecting local elasticity.



(a) Topography (PV=64nm) (b) Elasticity

Figure 2: A sensitivity-enhanced AFAM image captured for a titanium film, where a MC cantilever was adopted.

the mass ratio a ($=m_c/m_d$) should be larger than about 4 to enhance the sensitivity to the contact stiffness, where m_d is the distributed mass of a cantilever, to obtain the maximum sensitivity (Muraoka [4]). An adequate-sized particle was picked up from a pinch of pure tungsten powder (Deoxidized powder, WWE03PB, 53μm mesh pass, Kojundo Chemical Lab.) by a micropipette in 3-D micromanipulation under an optical microscope. The mass (m_c) was roughly estimated to be about 535ng from the volume and the density, which corresponds to a =11.5. The particle was placed with UV-curable adhesive (NOA81, Norland Products) on the reflective side (opposite to the tip side) of the cantilever near the free end in micromanipulation. Figure 3 shows a SEM image of the MC cantilever. For quantitative evaluations of sample elasticity, the exact value of m_c and the particle shape are not important, rather the fundamental resonant frequency of the MC cantilever free from a sample.

The tip used in the present study is made of silicon with an apex radius of about 10nm. It is coated with Ti/Pt film of 25nm in thickness, where titanium thin film is inserted between a silicon tip and platinum coating to improve adhesive strength of the coating. Such a ductile metallic coat contributes to prolonging lifetime of the tip, rather than brittle hard coat such as W_2C film fatally meeting with abrupt detachments. However the shape of apex is easily deformed and unstable. The flat-ended tip proposed (see Figure 4) was made by pressing the Ti/Pt coated tip onto a flat diamond sample with a contact force of 1μN, where the metallic coating was plastically deformed. The shape of a tip is stable and leads to relatively long lifetimes, in addition to simplifying the description of the contact stiffness.

2.2 Experimental setup

An atomic force microscope (AFM; SPI3700-SPA270, SII) was used with a few modifications for measurements of contact resonance spectra. Figure 5 shows an experimental setup. An external function generator (FS-2121, TOA) was employed to provide a sinusoidal excitation of precise frequencies from 0kHz to 1MHz, which was applied to the piezoelectric device 1 beneath a sample. The cantilever vibrations were measured by means of an optical lever technique using the position sensitive detector (PSD), which is a built-in function of SPA270. The signals of cantilever vibrations and the reference (excitation) were fed to a lock-in amplifier (LI5640, NF) trough a heterodyne down-converter (Frequency Extender 5571, NF). The function generator, the heterodyne down-converter, and the lock-in amplifier were controlled by a personal computer together with GP-IB interfaces, where a software VEE (Agilent Technologies) was adopted.

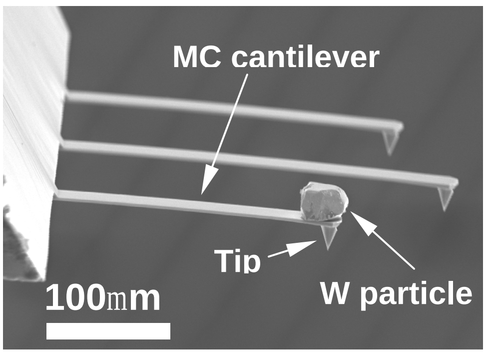


Figure 3: Mass-concentrated (MC) cantilever.

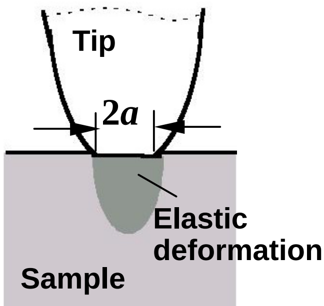


Figure 4: Flat-ended punch-like tip maintaining a constant contact area.

In advance of measurements of contact resonance, the free resonant frequencies were measured for the cantilever in no interaction with a sample. The measurements used a built-in piezoelectric device 2 in the cantilever holder. Then the cantilever was brought into contact with a sample. The contact force F_e was set several values from 100 nN to 500 nN for seeking a resonant peak sensitive to the contact stiffness. The time-averaged signal of cantilever deflection, i.e., the contact force, was maintained through a built-in feedback circuit for so-called contact mode, where the electronic circuit does not feel sinusoidal signals at ultrasonic frequencies. All the experiments were carried out at the temperature of 20°C and the relative humidity of about 40%.

Table 1: Bulk elasticity for standard samples.

Standard sample	Elastic coefficients E_i:Young's modulus n_i: Poisson's ratio	Effective Young's modulus
Diamond	E_{dia}=1050GPa n_{dia}=0.1	$E_{dia}/(1-n_{dia}^2)$= 1061GPa
Si(111)	E_{111}=187.9GPa n_{111}=0.180	$E_{111}/(1-n_{111}^2)$= 194.2GPa
Si(100)	E_{100}=130GPa n_{100}=0.278	$E_{100}/(1-n_{100}^2)$= 140.9GPa

A prerequisite for a standard sample is that the elastic coefficients are well defined for a surface layer of about 10nm in depth, where contact elastic deformation dominates for AFAM. We picked a diamond jewel, Si(111) and Si(100) wafers for standard samples. The silicon wafers were etched in saturated KOH solution for a few tens of hours to remove work-hardening layers or chemically modified layers. For these materials constituting of covalent bonds, interatomic forces act in the extremely short range, and loose and heterogeneous bands different form bulk nature are confined in very shallow surface layers such as a few atomic bonds in depth. Elastic coefficients of the standard surfaces are listed in Table 1.

3 THEORY ON A MC CANTILEVER EQUIPPED WITH A FLAT-ENDED

Flexural vibration of a cantilever having distributed mass exhibits infinite series of resonance. However the sufficient inertia of the concentrated mass degenerates all the resonant peaks for the deflection at the mass-attached site like a pinned edge, except a particular resonant peak. The resonance remained corresponds to that for translational motion of an effective mass connected with springs having no mass, as shown in Figure 6 (Muraoka [4]).

When the vibration amplitudes are small, the elastically deformed region near the contact

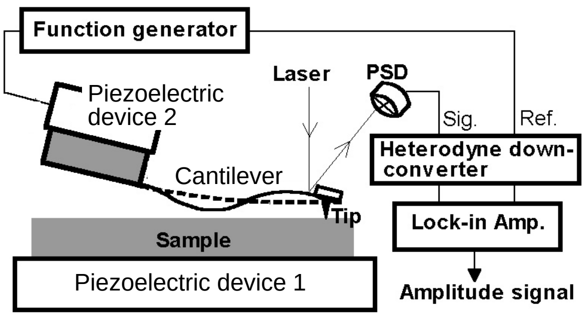


Figure 5: Experimental setup.

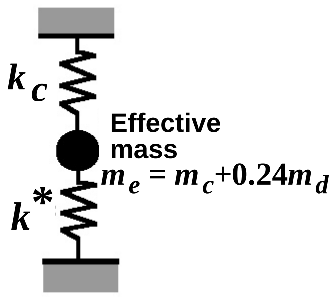


Figure 6: Spring-mass model.

area between a tip and a sample behaves as a linear spring, whose spring constant is called as the contact stiffness k^*. The resonant frequency f of MC cantilevers increases with k^* in accordance with the spring-mass model:

$$f = f_0\sqrt{1 + k^* / k_c} \quad , \tag{1}$$

where k_c (=1N/m) is a spring constant of the MC cantilever and f_0 is the fundamental resonant frequency of the MC cantilever free from a sample. Stiff samples like metal and ceramics take k^* =5x10^2 to 10^3 >>1 and then eqn (1) approximates to:

$$k^* / k_c = (f / f_0)^2 . \tag{2}$$

The intermolecular forces between the molecules constituting a tip and a sample and the meniscus forces arising from water film absorbed on a surface cause attractive force acting between a tip and a sample, i.e., adhesion force. Effects of the adhesion force on the contact stiffness cannot be ignored in conventional AFAM, where real profiles of tip apex are not simple and frequently assumed as spherical. The adhesion force influences the contact area as well as the contact force (F_e). In contrast, a flat-ended tip maintains a constant contact area independent of the adhesion force as well as of the contact force. The constancy ensures that the contact stiffness is also constant. A real flat-ended tip may have a slightly rough and rounded end-surface. Sufficient contact forces, however, realize the constant contact area. Then the following relation obtained from mechanics of linear elasticity (Nisitani [5]) is applicable for a flat-ended tip:

$$k^* = 2aE^* , \tag{3}$$

where a is a radius of the contact area and E^* is an effective Young's modulus defined as:

$$1 / E^* = (1 - \nu_t{}^2) / E_t + (1 - \nu_s{}^2) / E_s \quad , \tag{4}$$

where E_i and n_i are a Young's modulus and a Poisson's ratio, respectively. The indices i=t and i=s mean corresponding values for a tip and a sample, respectively.

4 RESULTS AND DISCUSSIONS

The optical lever technique built in the equipment detects directly the inclination of cantilever rather than the deflection. Therefore the amplitude signals measured contain trivial resonance peaks for vibration modes under a condition of pinned edge, in addition to the resonant peak resulting from translational motion of the concentrated mass, which corresponds to the resonant frequency (f) sensitive to the contact stiffness. The resonant frequencies under a condition of free end measured f=f_0= 9.304kHz for the fundamental resonance, 171.5kHz for the second resonance and 530.0kHz for the third resonance, where the latter two are trivial.

Figure 7 shows contact spectra observed for a standard sample of etched Si(100) wafer, where the contact force (F_e) was varied. Trivial resonant peaks of contact spectra must be close to those of free spectra (171.1kHz, 530.0kHz, …) because of the same modes (i.e., pinned edge). The guidance identified the resonance peak sensitive to the contact stiffness around 235kHz. When increasing F_e, the resonance peak sharpens to converge on a frequency of 236.1kHz. This confirms that the tip has a flat-ended profile. A contact force of F_e=500nN resulted in the reproducible resonance frequency f=f_{100}=236.1kHz. Figure 8 shows spectra measured under F_e=500nN for standard samples of Si(111) and diamond, which indicate the resonant frequencies f_{111}=255.4kHz

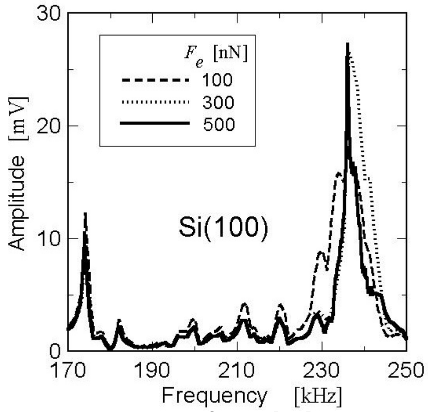


Figure 7: Spectra for etched Si(100) wafer.

Table 2: The present evaluations.

Effective Young's modulus of the tip	$E_t / (1- n_t^2) =$ 151.8GPa
Radius of the contact area	a = 4.39nm
Evaluated effective Young's modulus of etched Si(111) wafer	$E_{111}/(1- n_{111}^2) =$ 195.5GPa

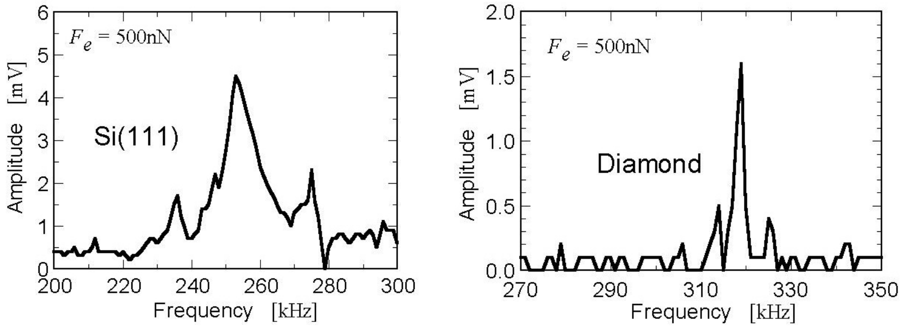


Figure 8: Spectra for etched Si(111) wafer and diamond.

and f_{dia}=318.7kHz, respectively.

Using eqns (2) to (4) along with the known values f_{100} , f_{dia} , $E_{100}/(1\text{-}n_{100}^2)$ and $E_{dia}/(1\text{-}n_{dia}^2)$, we inversely analyzed for a factor of A (=$a\, f_0^2/k_C$) and an effective Young's modulus $E_t/(1\text{-}\ n_t^2)$ for the tip, which are hard to be measured or estimated directly. It brought A=0.3800m/kg and $E_t/(1\text{-}n_t^2)$=151.8GPa. The latter is comparable to the averaged value for bulk platinum (196GPa) and bluk titanium (129GPa). Further use of k_c=1N/m and f_0=9.304kHz produced the reasonable contact radius a=4.39nm. Finally we examined the present method of evaluating sample elasticity for the precision. Combination of the measurement f_{111} and eqns (2) to (4) evaluated the effective modulus $E_{111}/(1\text{-}n_{111}^2)$=195.5GPa, which coincides with that for bulk Si(111) (194.2GPa) with an error of only 0.7 percent. The amazing example proves that the present method makes possible evaluations of local elasticity in a resolution of $2a$ = 9nm with high precision.

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