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Measuring AFM Cantilever Stiffness from a Thermal Noise Spectrum

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Abstract—The problem of AFM cantilever calibration from a thermal noise spectrum is considered. A large volume of preliminary work is required to use this method, along with consideration of numerous factors. Use of a synchronous detector allowed us to reduce the requirements for the measuring system and increase the accuracy of spring constant measurement. The respective experimental results are presented.

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INTRODUCTION

Atomic force microscopy (AFM) is now widely used all over the world. In high demand in various areas of knowledge such as microelectronics, defectoscopy, biology, and medicine, atomic force microscopy is considerably affecting the development of science as a whole. The importance of metrological measurements with atomic force microscopy has especially increased in recent years. An AFM probe is the most important part of any atomic force microscope [1]. The reliability and accuracy of metrological measurements depend to a great extent on how accurately the characteristics of the probe are determined while preparing for an experiment. The stiffness of the AFM cantilever or flexible console is one of the major quantitative characteristics of such a probe.

METHOD FOR DETERMINING AFM CANTILEVER STIFFNESS FROM A THERMAL NOISE SPECTRUM

Numerous methods are now known for measuring AFM cantilever stiffness, e.g., the Sader approach, static deflection, and calculating the spring constant from the geometric size of the flexible console [2–5]. Determining the AFM cantilever stiffness from a thermal noise spectrum is accepted as one of the most accurate and convenient methods [6]. It essentially includes the following. According to the equidistribution theorem, the energy of one degree of freedom is $0.5k_BT$, where $k_B = 1.3805 \times 10^{-23}$ J K^{-1} is the Boltzmann constant, and T is the absolute temperature. The equidistribution theorem assumes the following form for the first oscillation mode of a cantilever with spring constant k_{spr}

$$\left\langle \frac{1}{2} k_{spr} z^2 \right\rangle = \frac{1}{2} k_B T, \tag{1}$$

where z is the vertical deflection of the tip of a flexible console corresponding to the first oscillation mode. The expression for the spring constant from Eq. (1) is

$$k_{spr} = \frac{k_B T}{\left\langle z^2 \right\rangle}. \tag{2}$$

Expression $\langle z^2 \rangle$ can be replaced by the equivalent

$$\left\langle z^2 \right\rangle = \lim_{A \to \infty} \frac{1}{A} \int_0^A \left| z(t) \right|^2 dt. \tag{3}$$

We can use Fourier analysis to calculate this expression on the basis of experimental measurements. $\langle z^2 \rangle$ can be calculated using the following equation if N measurements of the deflection of the tip of flexible console z is conducted at equal intervals Δt:

$$\left\langle z^2 \right\rangle \approx \sum_{n=0}^{N/2} p_k \Delta f \approx \int_0^{f_c} p(f) df, \tag{4}$$

where $\Delta f = \frac{1}{N \Delta t}$, p_k is the power of a thermal noise spectrum at frequency $f_k = k\Delta f$, $f_c = \frac{1}{2\Delta t}$ is the Nyquist frequency, and $p(f)$ is a theoretical curve approximating the thermal noise power spectrum. The required response time of the measuring system can be estimated from (4). The Nyquist frequency must exceed the first resonance frequency of the AFM cantilever, and the number successively accumulated N values must provide resolution considerably greater than the width of the resonance peak:

$$\begin{cases} f_{samp} > 2 f_{res} \\ N \gg \dfrac{f_{samp}}{(f_{res}/Q)} \end{cases}. \tag{5}$$

The denotation in Eqs. (5) presenting the requirements for the measuring system is as follows: f_{samp} is frequency discretization, f_{res} is resonance frequency, and Q is the quality of AFM cantilever oscillation.

The spectrum is approximated by theoretical curve $p(f)$ in order to increase measurement accuracy, consider foreign noises, and confirm indirectly our model of AFM cantilever behavior. Virtually all of the area under the curve approximating the spectrum of thermal oscillation power z in the region of the AFM cantilever's first resonance frequency is used as $\langle z^2 \rangle$ in calculating the result.

LORENTZ MODEL

A Lorentz model describing the spectrum of the thermal oscillation power in the vicinity of the first resonance frequency by the following equation is usually used for an AFM cantilever in air:

$$p(f) = L(f) = \frac{a_0}{(f - a_1)^2 + a_2} + a_3, \quad (6)$$

where a_0, ..., a_3 are approximation parameters. Here, a_3 corresponds to foreign noise whose spectrum of which is assumed to be uniform and constant in the considered frequency range. The a_0, ..., a_2 parameters have the following physical significance: they are related to the height of resonance peak L_{peak}, AFM cantilever resonance frequency f_{res}, and quality factor Q:

$$f_{res} = a_1, \quad (7)$$

$$Q = \frac{a_1}{2\sqrt{a_2}}, \quad (8)$$

$$L_{peak} = \frac{a_0}{a_2} = \frac{2Qk_B T}{\pi k_{spr} f_{res}}. \quad (9)$$

Lorentz model (6) allows calculation of the integral (4)

$$\int_0^\infty \frac{a_0 df}{(f - a_1)^2 + a_2} \approx \int_{-\infty}^\infty \frac{a_0 df}{(f - a_1)^2 + a_2} = \frac{\pi a_0}{\sqrt{a_2}}. \quad (10)$$

Expression for the AFM cantilever spring constant assumes the form

$$k_{spr} = \frac{k_B T}{\pi a_0 / \sqrt{a_2}}. \quad (11)$$

Equation (9) can be used to determine the applicability of our method for a given instrument. The thermal oscillation peak must be distinguishable against the background of the measuring system's foreign noise.

USING A SYNCHRONOUS DETECTOR

Requirements (5) for the measuring system are critical for many existing instruments. The problem of an insufficiently rapid response time is aggravated even more when dealing with AFM cantilevers that have high resonance frequency and a high quality factor. To alleviate this problem, an original technical solution was proposed that allowed us obviate requirements (5).

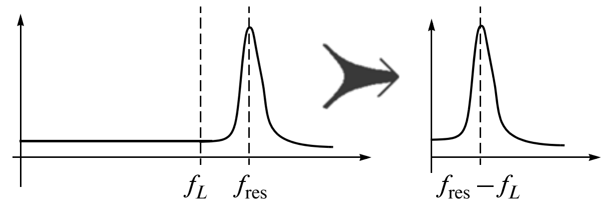

Fig. 1. Left: approximate spectrum of the input SD signal. Right: output SD signal spectrum.

Almost every modern atomic force microscope includes a synchronous detector (SD) used for determining the amplitude and phase of AFM cantilever oscillations [6]. The heart of the approach is using the SD for data acquisition. We assume that optical signal $s(t)$ corresponding to the vertical deflection of the tip of a flexible console is fed to the SD:

$$s(t) = \sum C_k \cos(2\pi f_k t + \theta_k), \quad (12)$$

where C_k are Fourier coefficients breaking the signal down into components $\cos(2\pi f_k t + \theta_k)$. The SD reference frequency is selected to be close to cantilever resonance frequency f_{res}. The reference SD signal takes the form

$$r(t) = R\cos(2\pi f_L t). \quad (13)$$

The SD output signal is thus $s(t)r(t)$, and the spectrum of the output signal is shifted relative to the spectrum of the input signal by reference frequency f_L (Fig. 1):

$$s(t)r(t) = \frac{R}{2}\sum C_k \cos(2\pi(f_k - f_L)t + \theta_k) + H(t). \quad (14)$$

Here $H(t)$ is a high frequency component that can be filtered out by the SD output filters, and instead of (5), we can write

$$\begin{cases} f_{samp} > 4 f_{res} Q \\ N \gg \dfrac{f_{samp}}{(f_{res}/Q)} \end{cases}. \quad (15)$$

The SD thus allows us to reduce the requirements for the measurement system response time.

RESULTS AND DISCUSSION

Measurements of AFM cantilever stiffness using a thermal noise spectrum were conducted on an Ntegra Prima (ZAO Nanotekhnologia-MDT, Zelenograd, Russia) scanning atomic force microscope using our software. Stiffness assessment was completely automated.

The measurements were performed with the SD as described above; the thermal noise spectrum was calculated using a fast Fourier transform (FFT). The results from approximating the NSG10 AFM cantilever spectrum using a Lorentz curve are presented in Fig. 2. The NSG10 spectrum was recorded in the following manner: The SD output signal was recorded in

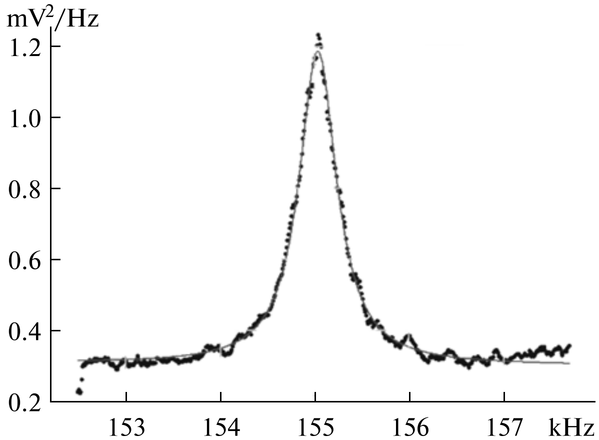

Fig. 2. Result from our NSG10 AFM cantilever spectrum approximation by a Lorentz curve.

series of $N = 1024$ values at the rate $f_{samp} = 15.6$ kHz, FFT were calculated for each series, and the obtained spectrum was averaged over the recorded series.

The optical system for registering the bending of the AFM probe's flexible console was calibrated by recording the traverse force curve of a smooth solid surface. The accuracy of stiffness measurement was 3–4% for AFM cantilevers with spring constants of up to 50 N m^{-1}.

CONCLUSIONS

The high accuracy of the thermal noise spectrum approach to measuring AFM cantilever stiffness was confirmed in our experiments. The precision of our Lorentz approximation of the thermal noise spectrum of an AFM probe in air was established. It was demonstrated that using an SD lowers the requirements for the measuring system.

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