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Cantilever optimization for applications in enhanced harmonic atomic force microscopy

Weijie Zhang, Yuhang Chen*, Jiaru Chu

Department of Precision Machinery and Precision Instrumentation, University of Science and Technology of China, Hefei 230027, China

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ABSTRACT

Structural design and optimization of atomic force microscopy (AFM) cantilevers were performed with multi-objectives to tailor one or more higher-order resonance frequencies to be integer multiples of the fundamental one and to keep the minimum change of stiffness. The tuning of frequency properties was achieved by altering mass distribution of the cantilever via cutting a rectangular slot. Rayleigh-Ritz method and finite element analysis were incorporated in the optimizations of slot dimension and position. The determined structure was fabricated on a conventional AFM cantilever by using focused ion beam etching. Higher harmonic imaging with the micromachined cantilever on a two-component polymer blend was subsequently performed. Experiments and theoretical simulations demonstrated that the harmonic amplitude contrast was improved up to at least 3 times. The enhancement of harmonic signals can benefit the discrimination of materials with different elastic properties.

1. Introduction

Dynamic atomic force microscopy (AFM) methods have been intensively adopted for nanoscale imaging and characterization [1]. In the most popular tapping mode, the probe tip driving at or near the fundamental resonance frequency can periodically touch the sample surface. Resulting from the nonlinear tip-sample interactions, higher harmonics of the base frequency of the oscillating cantilever emerge [2]. These harmonic responses are mainly dominated by contact forces and contact time. Consequently, they carry rich information on the mechanical properties of the specimen [3–5]. Theoretical analysis has demonstrated that the harmonic signals are very sensitive to the variation of local elasticity [6]. Therefore, harmonic amplitude images have higher spatial resolution than topography when biological samples are scanned [7,8]. In this regard, one of the most appealing characteristics of harmonic AFM is that it opens a way for fast, high-resolution and non-destructive mechanical mapping of various soft specimens including tumors, cells, polymers and biofilms [9–13].

In liquid, harmonic signals could be intrinsically large due to the low quality factor of the cantilever vibrations. Unfortunately, unsatisfactory signal-to-noise ratio will be encountered in the ambient environment [14]. For instance, higher harmonic amplitudes are usually several orders of magnitude smaller than that at the fundamental resonance frequency [3]. Therefore, there is an urgent demand in developing effective enhancement methods especially when acquiring harmonic images in air [12]. In terms of cantilever dynamics, if a higher harmonic is located at or near one of the resonance frequencies, the response at such a frequency can be strengthened. However, for most commercial cantilevers, their resonance frequencies of higher eigenmodes do not naturally equal to any harmonics of the fundamental resonance frequency.

Several approaches have been proposed to tune the frequency characteristics of a conventional AFM cantilever for better harmonic signals. Among them, perhaps the most convenient way is to change the mass distributions of the cantilever via either adding or removing local masses [15]. In such a way, a higher eigenmode resonance frequency can be adjusted to be an integer multiple of the first resonance frequency and subsequently it results in enhancement of the corresponding harmonic response. Following this approach, Sahin et al. matched the third flexural resonance frequency to the 16th harmonic of the fundamental one by selectively removing mass from the cantilever [16]. Li et al. attached a carefully positioned particle to tailor the second and the third eigenfrequencies simultaneously [17]. However, if a large frequency tuning ratio is required, the attached mass needs to be quite heavy. As a consequence, the fundamental frequency will be significantly lowered, which is undesirable in some situations. In addition, batch production may be difficult because both the mass and its attaching position need to be precisely controlled. Felts et al. introduced a

* Corresponding author.
E-mail address: chenyh@ustc.edu.cn (Y. Chen).

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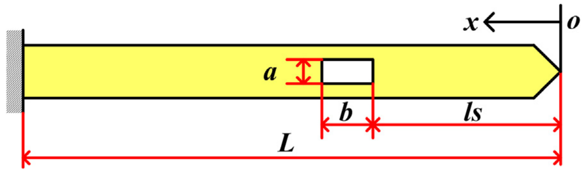

Fig. 1. Schematic illustration of the harmonic AFM cantilever. Structural design is based on a conventional rectangular cantilever with an additional cutting slot. *a* and *b* are respectively the slot width and length. *ls* denotes the starting position of the slot from the cantilever free end and *L* is the cantilever length.

paddle structure on the cantilever to adjust the ratio of the first two eigenfrequencies in a range from 1.6 to 4.5 [18]. However, the accompanied stiffness change will be large especially when a small frequency ratio is wanted and the design is also quite complicated. Cai et al. proposed two types of harmonic cantilevers, with variable-width and step cross-section respectively [19,20]. Such structures make the fabrication process rather complex and lead to difficult control of geometrical precision in manufacturing.

Even with all these progress, we should note that most previous harmonic cantilever modifications only focused on tuning the frequency characteristics while multiple objectives may be necessary for practical applications including requirements of stiffness and quality factor [21]. It has already been analyzed that the contact force and the contact time in each oscillation period are directly relevant with these physical quantities [22]. As a prototype, here we designed a cantilever with a cutting slot to satisfy both frequency and stiffness constraints. Based on Rayleigh-Ritz method and finite element analysis (FEA), theoretical approaches were employed to realize the structural optimization. After that, focused ion beam (FIB) etching was utilized to drill the slot on a conventional cantilever. Finally, harmonic AFM imaging using the modified cantilever was performed to ascertain the enhanced capability in discriminating materials with different elastic properties. It is worth mentioning that the cantilever structure is easily realizable for well-developed microfabrication techniques such as photolithography though FIB is applied in this work. As long as the slot dimension and position are finally determined, batch fabrications are feasible.

2. Harmonic cantilever optimization

The schematic illustration of the harmonic cantilever is presented in Fig. 1. Frequency tuning is realized by cutting out a rectangular slot on a common AFM cantilever to alter the mass distribution. The width *a* and length *b* of the slot together with its position *ls* are adjusted in the optimization processes. Parameters *ls* and *le* are the starting and ending positions of the slot, that is, $le = ls + b$. Note that the *x*-axis here is defined as starting from the free end of the cantilever. Multiple objectives are taken into consideration. First, in constraints of frequency features, f_2/f_1 and f_3/f_1 should be two integers with f_1, f_2 and f_3 respectively defined as the first, second and third eigenmode frequencies. This kind of characteristics enables enhancements of multiple harmonic signals. Second, for the demonstration of multi-objective optimization, the stiffness change Δk is kept minimum after the slot cutting. Further constraints such as the maximum allowable stress can also be included.

For a simple analysis, the cantilever geometry as illustrated in Fig. 1 is firstly approximated as an ideal rectangular shape with a cutting slot. The natural flexural vibrations of the cantilever beam is governed by,

$$EI\frac{\partial^4 y(x,t)}{\partial x^4} + \rho A\frac{\partial^2 y(x,t)}{\partial t^2} = 0 \tag{1}$$

Here $y(x,t) = z(x)e^{i\omega t}$ is the transverse displacement satisfying the boundary conditions that the deflection and slope at the clamped end are both zeros. ω is the angular frequency. E is Young's modulus of the cantilever material. I denotes the moment of inertia. ρ is the mass density and A is the section area. The damping is ignored, which has little influence on the eigenfrequencies in air. To solve the differential equation, Rayleigh-Ritz method is verified to be an easy approach [23]. In this method, the displacement of the beam is approximated in terms of a set of basis functions and unknown parameters,

$$z(x) = \sum_{i=1}^{n} \varphi_i z_i(x) = \sum_{i=1}^{n} \varphi_i \left(1 - \frac{x}{L}\right)^2 \left(\frac{x}{L}\right)^{i-1} \tag{2}$$

In the above equation, φ_i is a set of non-zero coefficients and the mode shape $z(x)$ satisfies the boundary conditions. Substituting Eq. (2) into Eq. (1) and applying the principle of minimum potential energy yield an eigenvalue problem [24],

$$\sum_{j=1}^{n} \left(k_{ij} - \omega^2 m_{ij}\right) \varphi_j = 0 \tag{3}$$

here the generalized mass m_{ij} and the generalized stiffness k_{ij} are expressed as,

$$m_{ij} = \int_0^L \rho A z_i(x) z_j(x)\,dx - \int_{ls}^{le} \rho A_c z_i(x) z_j(x)\,dx \tag{4}$$

$$k_{ij} = \int_0^L EI \frac{\partial^2 z_i(x)}{\partial x^2}\frac{\partial^2 z_j(x)}{\partial x^2}dx - \int_{ls}^{le} EI_c \frac{\partial^2 z_i(x)}{\partial x^2}\frac{\partial^2 z_j(x)}{\partial x^2}dx \tag{5}$$

In the above equations, A_c and I_c are respectively the section area and the moment of inertia of the slot structure. From Eq. (3), the angular eigenfrequencies ω can be obtained considering the relation that the following determinant equals zero,

$$\begin{vmatrix} k_{11} - \omega^2 m_{11} & k_{12} - \omega^2 m_{12} & \cdots & k_{1n} - \omega^2 m_{1n} \\ k_{21} - \omega^2 m_{21} & k_{22} - \omega^2 m_{22} & \cdots & k_{2n} - \omega^2 m_{2n} \\ \vdots & \vdots & & \vdots \\ k_{n1} - \omega^2 m_{n1} & k_{n2} - \omega^2 m_{n2} & \cdots & k_{nn} - \omega^2 m_{nn} \end{vmatrix} = 0 \tag{6}$$

Above theoretical analysis can be roughly used for optimization. However, for better approximations of the realistic AFM cantilever such as the non-ideal rectangular shape near the free end (see Fig. 1), FEA simulation will be much more accurate, in which real shape and dimension of the base cantilever can be modeled according to scanning electron microscopy (SEM) measurements.

3. Results and discussion

First, we try to obtain an initial view on the frequency tuning sensitivity, which is defined as the change of the ratio of a higher order eigenfrequency to the fundamental one per unit change of the slot position *ls* (see Fig. 1), that is, $S = \partial(f_n/f_1)/\partial ls$. This quantity is aimed to provide a general clue on the selection of suitable cutting positions. The typical results are demonstrated in Fig. 2. In these calculations, the density of the cantilever was 2330 kg/m^3, the elastic modulus was set as 170 GPa and the Poisson's ratio was 0.3. The main dimensional parameters were cantilever length of 465 μm, width of 50 μm and thickness of 2 μm. These parameters were chosen with respect to a commercial silicon cantilever (ContAl-G cantilevers, Budget Sensors). The slot length and width were kept as constants of 30 μm and 10 μm, respectively. Then, the slot position was varied from 30 to 430 μm with a step of 10 μm. For each cutting location, the corresponding eigenfrequencies were calculated using