

On the Determination of the Rigidity Parameters of Nanoobjects

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Abstract—The aim of this work is to develop theoretical grounds for experimental determination of the rigidity parameters of nanoobjects. An efficient way of determining elastic moduli used in macromechanics consists in measuring the eigenfrequencies of an object under test. Details of applying this approach to nanoobjects are discussed. A method of experimental determination of the rigidity parameters is suggested that is based on dynamic damping of vibrations (antiresonance). This method provides the possibility of separating out the eigenfrequencies of the nanoobject from the frequency spectrum of a system composed of a nanoobject and the cantilever of an atomic force microscope.

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INTRODUCTION

Interpretation of experimental data is a challenging scientific problem. This problem becomes still more acute in studying nanometer-scale objects. This is because experimental conditions for nanoobjects differ radically from those for macroobjects. In the latter case, the dimensions of measuring devices (e.g., strain gages) are, as a rule, much less than those of an object being studied. Therefore, in experiments with macroobjects, measuring equipment has a negligible effect, if any, on the object and measurements give its true characteristics. In the case of nanoobjects, the situation is basically different, since microinstruments used in these experiments may strongly influence the object, changing its properties up to restructuring or even causing destruction. Eventually, measurements may represent the characteristics of the modified nanoobject or of the nanoobject–instrument system. Thus, to study the interplay between nanoobjects and measuring equipment is of great importance. Below, this problem is exemplified in determining the elastic properties of nanoobjects with an atomic force microscope (AFM).

DETERMINATION OF THE ELASTIC MODULI OF NANOOBJECTS

The problem of finding the elastic moduli of nanometer-scale objects has become topical. Many research-

ers note that the values of elastic moduli derived from micro- and macroexperiments differ. In [1, 2], the Young's modulus and flexural rigidity of a 2D single-crystal sheet was studied as a function of the number of atomic layers in the sheet. The results obtained in [1, 2] indicate that three expressions for the flexural rigidity of the sheet (the expression known from the continuum theory, the expression obtained by substituting Young's modulus into the expression from the continuum theory, and that based on a discrete model [2]) give results differing considerably for a small number of atomic layers. This means that the formulas from the continuum theory, which ignore the discreteness of the material properties across the thickness of the sheet, may yield erroneous results. Clearly, the discreteness along the length of the sheet, where the number of layers is large, is insignificant and the continuum expressions seem to apply well in this case. The same is true for analysis of nanorods and nanosheaths. Thus, it is important to devise techniques allowing for directly finding the elastic properties of thin-walled nanoobjects, i.e., without using formulas relating the elastic moduli of a nanoobject to its thickness and the Young's modulus of the material. Specifically, a burning issue is experimental determination of the mechanical properties of nanoobjects [3].

An efficient way of determining elastic moduli, which are used in macromechanics, consists in measur-

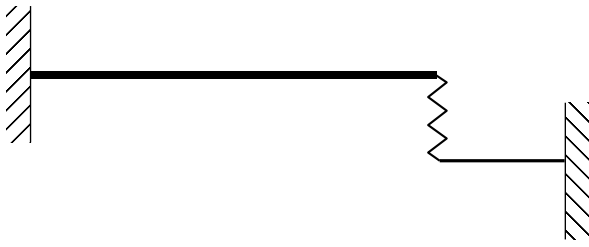


Fig. 1. Cantilever (at the left) and nanorod (at the right).

ing the eigenfrequencies of an object under test (the resonance method). Below, details of applying the resonance method to nanoobjects are discussed and another (antiresonance) method is suggested.

AFM MEASUREMENT OF THE NANOOBJECT'S VIBRATION EIGENFREQUENCIES

Today, the properties of nanoobjects, including their eigenfrequencies, are studied with probe microscopy, specifically, with an AFM, which interacts with a test object through an electric field [4, 5]. The major component of the AFM is a scanning probe (cantilever) [6, 7]. Mechanically, the cantilever represents an elastic beam one end of which is tightly fixed and the other is free and bears a nanometer tip with a radius of curvature of 10–50 nm. Standard commercial cantilevers measure $200 \times 35 \times 1.5 \mu\text{m}$ and have resonance frequencies ranging from 10 to 400 kHz.

The AFM operates in three (contact, contactless, and tapping) modes. Note that all the modes are contact from the mechanical point of view. As the tip approaches a test object, they come into interaction, no matter whether this is interaction is contact or is accomplished through force fields. The dependence of the force of interaction on the tip–object distance is akin to that observed in the case of interaction with the Lenard–Jones potential. Depending on the tip–object distance, the force of interaction is either repulsive (contact mode), attractive (contactless modes), or alternating (tapping mode). All the three modes can be used to examine the surface relief, and the tapping mode is also applied to determine the eigenfrequencies. In this mode, the clamped end of the cantilever executes vertical vibrations with a given frequency. Measuring the vibration amplitudes of cantilever points at different frequencies, one fixes resonances. If the tip is away from the surface, the resonance frequencies are close to the eigenfrequencies of the cantilever. As the tip approaches the surface and comes into interaction with it, the resonance frequencies vary. From the dependence of the resonance frequency on the tip–surface distance, one can judge the properties of the test object.

Application of an AFM to measure eigenfrequencies is not free of disadvantages and limitations, which do not allow making good use of the potential of this

instrument. The problems associated with this method are the following.

(1) The frequency range accessible to measurements is limited. To extend it to higher frequencies requires a decrease in the dimensions (weight) of the cantilever and/or an increase in its rigidity.

(2) Imperatively, the top of the tip must be much smaller than the test object; therefore, the radius of curvature of the tip should be decreased as the test object gets smaller.

(3) The nanoobject should be mounted so that the support has no influence on its eigenfrequencies. Otherwise, extra difficulties in interpreting measurement results and gaining information on the nanoobject may arise.

(4) The tip is in contact with the object and inevitably influences it somehow or other. As a result, the eigenfrequencies of the nanoobject–cantilever system, rather than of the nanoobject itself, are measured.

The last-mentioned problem is related to the well-known mechanical effect: the redistribution of the vibration eigenfrequencies of the cantilever–object system among the cantilever and object [8]. In this case, the change in the frequency spectrum considerably depends on the tip–surface distance, since such a redistribution is equivalent to a change in the “rigidity” of field interaction coupling.

Physically, two problems lying at the interfaces between mechanics and experimental physics arise here: (i) determination of the nanoobject's elastic moduli from the eigenfrequencies of the system and (ii) design of experiments making it possible to separate out the eigenfrequencies of a nanoobject from the frequency spectrum of the system.

SYSTEM UNDER TEST

The problem stated here and the cantilever–nanoobject test system elaborate upon work [6]. The basic difference is that here test nanoobjects possess intrinsic dynamics. Examples are the elements of thin-walled (nanodimensional) structures, such as rods, sheaths, and helices. Below, we set forth theoretical grounds for determining the vibration eigenfrequencies of rodlike structures with an AFM.

Consider the mechanical model of a test object shown in Fig. 1. The rod at the left of the figure symbolizes a cantilever. Its left end is firmly fixed, while the right end interacts with the object. The bend of the cantilever in the vertical direction is specified by function $u(x_1, t)$, where x_1 is the coordinate of the rod along its length (at the left end of the rod, $x_1 = 0$). Let L_1 , D_1 , and ρ_1 designate the length, flexural rigidity, and linear density of the cantilever, respectively. Rigidity C_1 of the cantilever, which is usually included in specifications, is defined as the force acting upon the tip divided by the displacement of the free end of the cantilever. It is easy

to show that rigidity C_1 and flexural rigidity D_1 of the cantilever are related to each other as $C_1 = \frac{3D_1}{L_1^3}$. The

nanorod at the right simulates an object the flexural rigidity of which is to be found [9, 10]. Its right-hand end is firmly fixed, and the left-hand one interacts with the cantilever. The vertical bend of the nanorod is given by function $v(x_2, t)$, where x_2 is the coordinate of the nanorod along its length (at its right-hand end, $x_2 = 0$). Let L_2, D_2 , and ρ_2 designate the length, flexural rigidity, and linear density of the nanorod, respectively. Cantilever-rod field interaction is simulated by linear rigidity C of the spring, which, in essence, means linearization of the Lenard-Jones (or any other) interaction potential in the static equilibrium state. It should be noted that the position of static equilibrium is easy to find in experiments. Away from the surface being studied, the cantilever is in the horizontal position. Approaching to the surface, it starts deforming but takes the horizontal position again at some distance to the surface, signifying static equilibrium. At the zero time, the rods are assumed to be undeformed and the spring unstrained. At static equilibrium, the tip-object coupling rigidity (spring rigidity) is high; so, $C \gg C_1$. For this reason, to find coupling rigidity C from static experiments is a great challenge: the difference between the displacements of the tip and surface is within the measurement error. Measurements in the ranges $C \sim C_1$ and $C \ll C_1$ are difficult to take, since these ranges correspond to the uncertain portion of the force-displacement diagram.

FREE VIBRATIONS OF THE CANTILEVER-NANOOBJECT SYSTEM

The basic equations of dynamics that describe the free vibrations of the given mechanical system have the form

$$D_1 u^{IV} + \rho_1 \ddot{u} = 0, \quad D_2 v^{IV} + \rho_2 \ddot{v} = 0. \quad (1)$$

They are complemented by the boundary conditions

$$\begin{aligned} u(0) = 0, \quad u'(0) = 0, \quad u''(L_1) = 0, \\ D_1 u'''(L_1) = C[u(L_1) - v(L_2)], \\ v(0) = 0, \quad v'(0) = 0, \quad v''(L_2) = 0, \\ D_2 v'''(L_2) = -C[u(L_1) - v(L_2)]. \end{aligned} \quad (2)$$

The spectral problem meeting Eqs. (1) is stated in line with finding the vibration eigenfrequencies of the system; that is, a solution is sought in the form $u(x_1, t) = u_0(x_1)e^{i\omega t}$, $v(x_2, t) = v_0(x_2)e^{i\omega t}$. Solving this spectral

problem yields the frequency equation

$$\begin{aligned} [1 + \cos(\lambda L_1) \cosh(\lambda L_1)] & \left(1 + \cos(\mu L_2) \cosh(\mu L_2) \right. \\ & \left. + \frac{C}{D_2 \mu^3} [\sin(\mu L_2) \cosh(\mu L_2) - \cos(\mu L_2) \sinh(\mu L_2)] \right) \\ & + \frac{C}{D_1 \lambda^3} [\sin(\lambda L_1) \cosh(\lambda L_1) - \cos(\lambda L_1) \sinh(\lambda L_1)] \\ & \times (1 + \cos(\mu L_2) \cosh(\mu L_2)) = 0, \end{aligned} \quad (3)$$

where

$$\lambda^2 = \sqrt{\frac{\rho_1}{D_1}} \omega, \quad \mu^2 = \sqrt{\frac{\rho_2}{D_2}} \omega, \quad (4)$$

and ω is the eigenfrequency of the system. As follows from (3), the eigenfrequencies depend on all the parameters of the system, so that those of the nanorod are impossible to separate out from the frequency spectrum of the system. Lets us find a relationship between quantities $\frac{C}{D_1 \lambda^3}$ and $\frac{C}{D_2 \mu^3}$. Making use of formulas (4) for the wavenumbers and expanding known quantities $D_i = E_i I_i$ and $\rho_i = \rho_i^* S_i$ (E_i are the Young's moduli of the rods, ρ_i^* are their volume densities, and I_i and S_i are the moments of inertia and cross-sectional areas of the rods; $i = 1, 2$), we obtain

$$\begin{aligned} D_1 \lambda^3 &= (\rho_1^* S_1)^{3/4} (E_1 I_1)^{1/4} \omega^{3/2}, \\ D_2 \mu^3 &= (\rho_2^* S_2)^{3/4} (E_2 I_2)^{1/4} \omega^{3/2}. \end{aligned} \quad (5)$$

If the rods have $h_i \times a_i$ rectangular cross sections, where h_i and a_i are, respectively, their thicknesses and widths, expressions (5) take the form

$$\begin{aligned} D_1 \lambda^3 &= (\rho_1^*)^{3/4} \left(\frac{E_1}{12} \right)^{1/4} a_1 (h_1 \omega)^{3/2}, \\ D_2 \mu^3 &= (\rho_2^*)^{3/4} \left(\frac{E_2}{12} \right)^{1/4} a_2 (h_2 \omega)^{3/2}. \end{aligned} \quad (6)$$

For rods of arbitrary cross section, the following estimates are valid:

$$\begin{aligned} D_1 \lambda^3 &\sim (\rho_1^*)^{3/4} \left(\frac{E_1}{12} \right)^{1/4} d_1^{5/2} \omega^{3/2}, \\ D_2 \mu^3 &\sim (\rho_2^*)^{3/4} \left(\frac{E_2}{12} \right)^{1/4} d_2^{5/2} \omega^{3/2}, \end{aligned} \quad (7)$$

where d_i are the characteristic linear sizes of the cross sections. From (6) and (7), we have

$$\frac{C}{D_1 \lambda^3} \ll \frac{C}{D_2 \mu^3}. \quad (8)$$

Thus, frequency equation (3) can be approximated as

$$[1 + \cos(\lambda L_1) \cosh(\lambda L_1)](1 + \cos(\mu L_2) \cosh(\mu L_2) + \frac{C}{D_2 \mu^3} [\sin(\mu L_2) \cosh(\mu L_2) - \cos(\mu L_2) \sinh(\mu L_2)]) = 0. \quad (9)$$

Equation (9) has two spectra of eigenfrequencies. The first one contains the vibration eigenfrequencies of the cantilever,

$$1 + \cos(\lambda L_1) \cosh(\lambda L_1) = 0; \quad (10)$$

the other, those of the nanorod with the spring-loaded end,

$$1 + \cos(\mu L_2) \cosh(\mu L_2) + \frac{C}{D_2 \mu^3} [\sin(\mu L_2) \cosh(\mu L_2) - \cos(\mu L_2) \sinh(\mu L_2)] = 0. \quad (11)$$

Thus, we managed to identify (separate out) the spectra for either rod, even having made not too strong assumption (8).

Note that $\mu L_2 \sim 1$ for the first eigenfrequencies of the nanoobject. Then, dimensional quantity $\frac{C}{D_2 \mu^3}$ can be estimated as

$$\frac{C}{D_2 \mu^3} \equiv \frac{C L_2^3}{D_2 (\mu L_2)^3} \sim \frac{C}{C_2}, \quad C_2 = \frac{3 D_2}{L_2^3}. \quad (12)$$

Since $C \gg C_2$, the inequality $\frac{C}{D_2 \mu^3} \gg 1$ is valid.

Consequently, Eq. (11) can be approximated as

$$\sin(\mu L_2) \cosh(\mu L_2) - \cos(\mu L_2) \sinh(\mu L_2) = 0. \quad (13)$$

Equation (13) represents the vibration spectrum of a rod one end of which is firmly fixed and the other is hinged.

The natural problem arises as to how to find the cantilever vibration waveform at the frequencies found above, since the cantilever vibration waveform has a considerably effect on the measurement accuracy when resonance is detected with a laser beam, which gives a spot of a certain size.

FORCED VIBRATIONS OF THE SYSTEM

Consider the forced harmonic vibrations of the system. Let

$$u(0, t) = A \sin(\Omega t), \quad A = \text{const.} \quad (14)$$

$$u(x_1, t) = [P_1 \cos(\lambda_* x_1) + P_2 \sin(\lambda_* x_1)$$

Solutions to the problem thus stated are functions

$$+ P_3 \cosh(\lambda_* x_1) + P_4 \sinh(\lambda_* x_1)] \sin(\Omega t), \quad (15)$$

$$v(x_2, t) = [Q_1 \cos(\mu_* x_2) + Q_2 \sin(\mu_* x_2) + Q_3 \cosh(\mu_* x_2) + Q_4 \sinh(\mu_* x_2)] \sin(\Omega t),$$

where $\lambda_*^2 = \sqrt{\frac{\rho_1}{D_1}} \Omega$ and $\mu_*^2 = \sqrt{\frac{\rho_2}{D_2}} \Omega$. Constants P_i and Q_i are found from the boundary conditions (the respective expressions are very awkward and therefore omitted). Notably, the denominators of the expressions for P_i and Q_i vanish when forced vibration frequency Ω coincides with one of the eigenfrequencies of the system, ω_n , given by (3). At $\Omega = \omega_n$, the cantilever vibration amplitude becomes indefinitely large in our model and sharply grows in real experiment. This fact allows us to find resonance frequencies coincident with the eigenfrequencies of the system.

DYNAMIC DAMPING OF VIBRATIONS

Experimentally, one can detect not only a sharp buildup of the vibration amplitude but also vanishing of the amplitude. In many-body systems with distributed parameters, the latter fact may be observed in two cases: (i) when the point where the amplitude is measured is a node of a given waveform of vibration or (ii) when the vibrations of one body are dynamically damped at the partial frequency of another (so-called antiresonance). Let us pose the question: whether there exist forced vibration frequencies Ω at which the cantilever's right-hand end, which is contact with the nanoobject, remains stationary at any time instant? One can answer this question having solved the equation

$$u(L_1, t) = 0. \quad (16)$$

Substituting expression (15) for $u(x_1, t)$ into (16), taking into account expressions for constants P_i and Q_i , and carrying out simple rearrangements, we obtain the equation

$$A D_1 D_2 \lambda_*^2 \mu_*^3 [2 \cosh(\lambda_* L_1) \sin(\lambda_* L_1) + 2 \sinh(\lambda_* L_1) \cos(\lambda_* L_1) + \sinh(2 \lambda_* L_1) + \sin(2 \lambda_* L_1)] \times (1 + \cos(\mu_* L_2) \cosh(\mu_* L_2) + \frac{C}{D_2 \mu_*^3} [\sin(\mu_* L_2) \cosh(\mu_* L_2) - \cos(\mu_* L_2) \sinh(\mu_* L_2)]) = 0. \quad (17)$$

Solving (17), one can find frequencies Ω_n at which the vibration amplitude of the cantilever's right end vanishes. It is easy to check that (17) can be split into

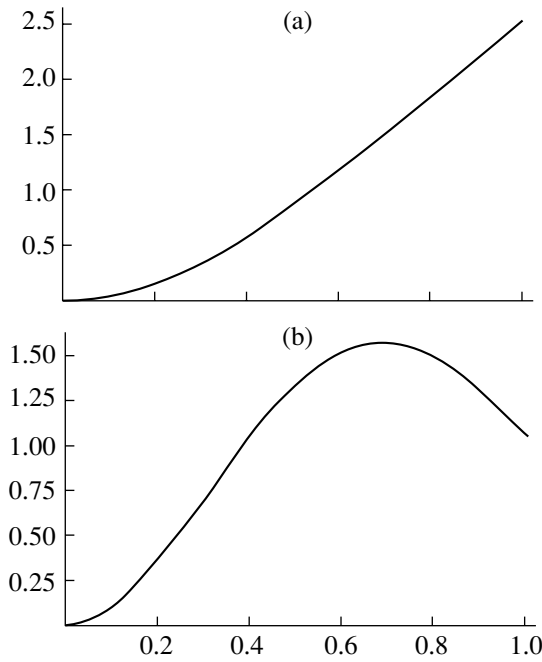


Fig. 2. Resonance waveforms ($L_2/L_1 = 1, h_2/h_1 = 1$).

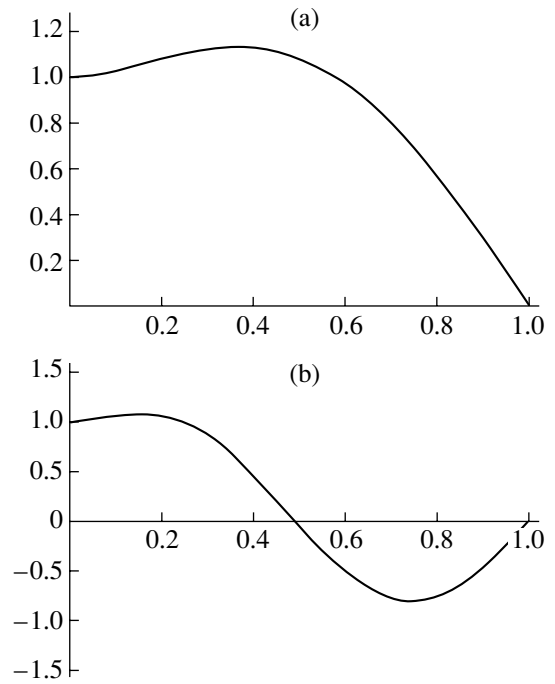


Fig. 3. Antiresonance waveforms ($L_2/L_1 = 1, h_2/h_1 = 1$).

two independent equations. The first one has the form

$$2 \cosh(\lambda_* L_1) \sin(\lambda_* L_1) + 2 \sinh(\lambda_* L_1) \cos(\lambda_* L_1) + \sinh(2\lambda_* L_1) + \sin(2\lambda_* L_1) = 0; \tag{18}$$

the other,

$$1 + \cos(\mu_* L_2) \cosh(\mu_* L_2) + \frac{C}{D_2 \mu_*^3} [\sin(\mu_* L_2) \cosh(\mu_* L_2) - \cos(\mu_* L_2) \sinh(\mu_* L_2)] = 0. \tag{19}$$

Equation (18) depends on only the cantilever parameters and so is of no interest for us, while Eq. (19) depends on the parameters of the nanorod and rigidity of nanorod–cantilever coupling. It is this equation that specifies “antiresonance” frequencies, at which the vibration of the cantilever’s right end is damped. It should be noted that Eq. (19) exactly coincides with Eq. (11), which specifies the eigenfrequencies of the hinged nanorod. Since Eq. (11) has been derived from the frequency equation by neglecting small quantities on the order of $\frac{C}{D_1 \lambda^3}$, it can be argued that antiresonance frequencies Ω_n are close to eigenfrequencies Ω_n of the system, differing from them by small quantities of the above order.

VIBRATION WAVEFORMS

Figures 2–5 show the first (panels “a”) and second (panels “b”) forms of cantilever vibrations at resonance (Fig. 2) and antiresonance (Figs. 3–5). The vertical axis in these figures plots the displacement of cantilever points; the abscissa axis, dimensionless coordinate x_1/L_1 . It is assumed that the cantilever and nanorod have the same sizes. When the nanorod diminishes in size, qualitatively the cantilever waveforms change insignificantly. The resonances are easy to detect with an AFM; the only and essential drawback here is that the resonance frequencies characterize the cantilever–test object system instead of the object itself. In light of this, the effect of antiresonance comes into importance, since it allows one to determine the vibration eigenfrequencies of the test nanostructure. The cantilever vibrations at antiresonance frequencies have a multinodal waveform. The number of nodes depends on the serial number of the waveform and on parameter

$$\frac{\mu L_2}{\lambda L_1} = \sqrt[4]{\frac{D_1 \rho_2 L_2}{D_2 \rho_1 L_1}} = \sqrt[4]{\frac{E_1 \rho_2^* I_1 S_2 L_2}{E_2 \rho_1^* I_2 S_1 L_1}}. \tag{20}$$

If the rods have a rectangular cross section $h_i \times a_i$, then

$$\frac{\mu L_2}{\lambda L_1} = \sqrt[4]{\frac{E_1 \rho_2^*}{E_2 \rho_1^*}} \sqrt{\frac{h_1 L_2}{h_2 L_1}}. \tag{21}$$

For rods of an arbitrary cross section with character-

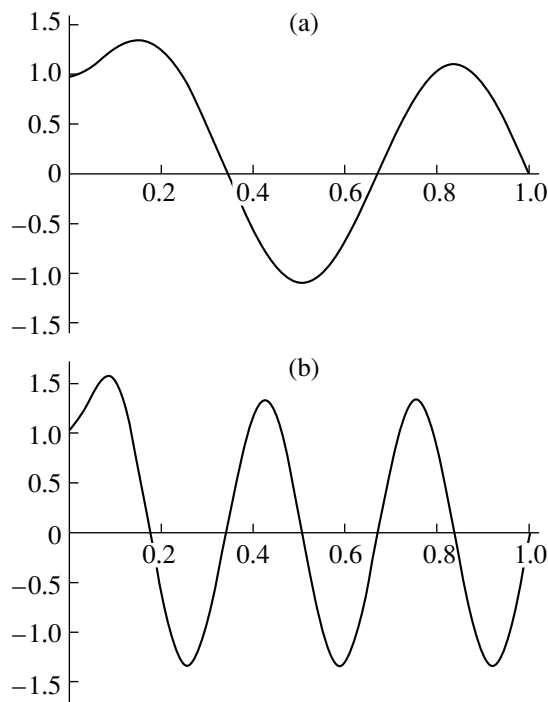


Fig. 4. Antiresonance waveforms ($L_2/L_1 = 0.1, h_2/h_1 = 0.1$).

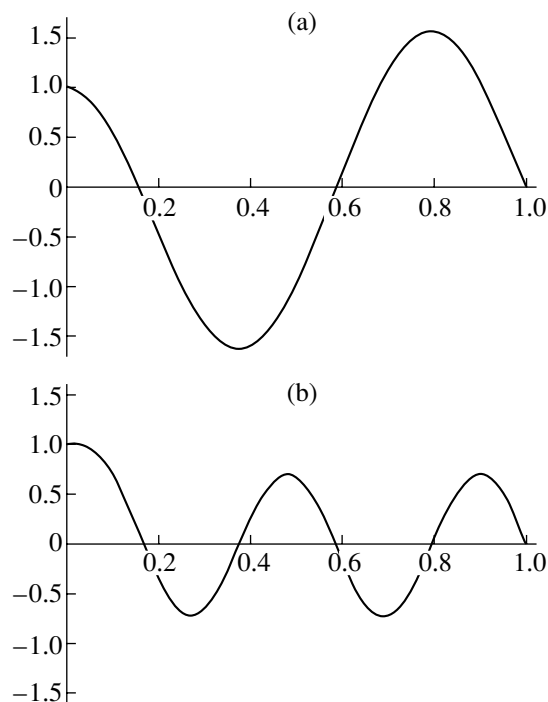


Fig. 5. Antiresonance waveforms ($L_2/L_1 = 0.04, h_2/h_1 = 0.01$).

istic linear sizes d_i , the estimate

$$\frac{\mu L_2}{\lambda L_1} \sim \sqrt[4]{\frac{E_1 \rho_2^*}{E_2 \rho_1^*} \frac{d_1 L_2}{d_2 L_1}} \quad (22)$$

is valid. If the cantilever and nanorod are of the same size, the first waveform of the cantilever has no nodes (Fig. 3a) and the second waveform has a single node (Fig. 3b). As the test nanorod is scaled down by a factor of 10, the antiresonance frequencies increase by the same factor and the waveforms of the cantilever become multinodal (Fig. 4). The antiresonance frequencies may increase to the point where they go beyond the measurement range of the instruments. When trying to detect an antiresonance in a multinodal waveform, one may run into obstacles associated with the fact that the laser beam used in the optics detecting the cantilever displacement [11] has a finite size and so the amplitude is averaged over a certain area of the rod rather than being measured at a point. If the length of the rod decreases not as strongly as the linear sizes of its cross section, the eigenfrequencies and number of nodes in the waveform grow with a lower rate. To make sure of it, it suffices to compare Fig. 4, which meets the case when all the linear sizes were decreased in proportion, with Fig. 5, which corresponds to the case when the linear sizes shrink more significantly but disproportionately. Thus, with a relationship between the cantilever and nanorod parameters chosen properly, the laser devices currently available can be used to detect antiresonances in the cantilever vibration waveforms.

CONCLUSIONS

The problem of finding the rigidity of nanodimensional objects was considered in [12]. In our case, the flexural rigidity of the nanorod can be found from both the resonance frequencies, using (3), and antiresonance frequencies using (19). Both equations involve two unknown parameters: flexural rigidity D_2 of the nanorod and rigidity C of cantilever–nanorod coupling (mass m_2 and length L of the cantilever can be measured, and its linear density ρ_2 can then be found by the formula $\rho_2 = m_2/L_2$). Having measured two (resonance or antiresonance) frequencies, one substitutes them into the respective equation, (3) or (19) and thus reduces the problem of finding the flexural rigidity of the nanorod to the solution of transcendental equations in two unknowns. Note that Eq. (19) is simpler than (3) and, unlike (3), does not contain small parameter $\frac{C}{D_1 \lambda^3}$.

Thus, determination of the flexural rigidity of the nanorod from antiresonance frequencies is computationally easier. However, it makes sense to apply both approaches and compare the obtained values of D_2 and C to improve the reliability of the results.

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