Method for the calibration of atomic force microscope cantilevers

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(Received 2 December 1994; accepted for publication 27 March 1995)

The determination of the spring constants of atomic force microscope (AFM) cantilevers is of fundamental importance to users of the AFM. In this paper, a fast and nondestructive method for the evaluation of the spring constant which relies solely on the determination of the unloaded resonant frequency of the cantilever, a knowledge of its density or mass, and its dimensions is proposed. This is in contrast to the method of Cleveland et al. [Rev. Sci. Instrum. 64, 403 (1993)], which requires the attachment of masses to the cantilever in the determination of the spring constant. A number of factors which can influence the resonant frequency are examined, in particular (i) gold coating, which can result in a dramatic variation in the resonant frequency, for which a theoretical account is presented and (ii) air damping which, it is found, leads to a shift of ~4% in the resonant frequency down on its value in a vacuum. Furthermore, the point of load on the cantilever is found to be extremely important, since a small variation in the load point can lead to a dramatic variation in the spring constant. Theoretical results that account for this variation, which, it is believed will be of great practical value to the users of the AFM, are given. © 1995 American Institute of Physics.

I. INTRODUCTION

Atomic force microscopy has rapidly emerged as a very general technique for atomic imaging of surfaces and for the measurement of interfacial forces. In the latter case, the deflection of a microcantilever is recorded as a function of the separation between the cantilever and a substrate, by monitoring the deflection of a laser beam reflected off the back of the cantilever, with a split photodiode. The force is then determined via Hooke's law. However, it must be emphasized that the accuracy of the force-distance data is predicated on an accurate knowledge of the spring constant of the cantilever. Therefore, the knowledge of the deflection properties of these cantilevers is of fundamental importance in the application of the atomic force microscope (AFM), as was discussed in Ref. 3. The use of V-shaped cantilevers is currently popular because they are less prone to lateral twisting and rolling as the cantilever jumps into and out of contact with the surface. Whereas a number of approximations exist relating the spring constant of a thin rectangular plate to its dimensions and Young’s modulus,2-3 until recently,4 no simple and accurate approximations existed for V-shaped cantilevers, and it has been customary to use the simple “two-beam” approximation, presented in Ref. 5. Recently, Sader and White presented the results of a finite element analysis of the static deflection of V-shaped cantilevers, which clearly demonstrated the inaccuracy of the two-beam approximation.5 They presented exact numerical results for the spring constants of V-shaped cantilevers with a variety of dimensions and Poisson ratios, which encompass cantilevers currently being employed in the AFM. It should also be noted that their results directly depend on the Young's modulus of the cantilever, as do the analytic results in Refs. 2-5. Unfortunately, microfabrication techniques often result in variable stoichiometry of the cantilevers and this, in turn, causes considerable variation in the elastic properties, in particular, the Young’s modulus. It is therefore unlikely that the bulk values, which themselves vary by some 20%, are applicable to microfabricated cantilevers,6 and this restricts the applicability of Sader and White’s results and other formulas which depend on Young’s modulus,2-5 unless reliable values for the elastic properties can be independently determined.

There are currently many methods available for the determination of the spring constants of AFM cantilevers (e.g., Refs. 7-10). These include the measurement of the static deflection of the cantilever,7,8 as well as dynamic measurements9,10 in the determination of the spring constants. Recently, Cleveland et al.10 proposed a method, henceforth referred to as the Cleveland method, for the determination of the spring constants of arbitrarily shaped thin cantilevers which involved the placement of spheres of different masses at the tip of the cantilever and measurement of its resultant resonant frequency. This therefore eliminated the necessity for the determination of Young’s modulus. However, due to the necessity for the attachment of spheres of differing mass to the cantilever, the method suffers from some practical difficulties which limit its accuracy, which we shall discuss in detail. In this paper, we also eliminate the necessity for the evaluation of Young’s modulus, necessary for use of the results in Refs. 2-5, and overcome the problems associated with the Cleveland method, by proposing a further method for the evaluation of the spring constant of arbitrarily shaped thin cantilevers. This method relies on the determination of the unloaded resonant frequency of the cantilever, which is a readily accessible quantity. This eliminates the necessity for the careful placement of spheres on the cantilever, as required by the Cleveland method, which we
shall also discuss. The resonant frequency of an AFM cantilever can be directly measured, by monitoring its vibrations due to thermal fluctuations, using a spectrum analyzer. We shall show theoretically, via an exact scaling argument which is applicable to arbitrary thin cantilevers, that the spring constant of the cantilever is directly related to its mass and resonant frequency by an exceedingly simple relation that is independent of Young’s modulus. This enables the spring constant of the cantilevers to be determined quickly and non-destructively, once the resonant frequency is known. We have found that with a knowledge of its dimensions, the spring constant of a single V-shaped cantilever may be determined within a few minutes. Furthermore, the resonant frequency is very sensitive to flaws or microcracks in the cantilevers, and its measurement readily shows whether the cantilever being employed is damaged.

In order to demonstrate the validity and accuracy of the proposed method, we shall present the results of an experimental investigation of the theoretical formulation. This shall involve in part a comparison with the Cleveland method. We also investigate the effect of air damping on the resonant frequency of the cantilevers, from which we find a significant shift in the resonant frequency as compared to the value in vacuum. The effect of load position on the cantilever is also investigated, from which it is found that a small shift in its position along the cantilever can result in a dramatic variation in the actual spring constant, and we present theoretical results for its correction. Finally, the effects of gold coating on the cantilevers are examined. In practice, gold coating is used to increase the reflectivity of the cantilevers. Due to the high density of gold, we find that its effect on the proposed method is dramatic and cannot be neglected. However, we are able to model this trivially and we present a theoretical account.

II. THEORY

We shall begin by presenting the background preliminary theory, pertinent to the proposed method. For a more comprehensive discussion the reader is referred to Refs. 2, 11, and 12.

It is well known that the governing equation for the dynamic deflection of a thin plate exhibiting small deflections $w$ is given by

$$D\nabla^4 w(x,y,t) + \sigma \frac{\partial^2 w(x,y,t)}{\partial t^2} = q(x,y,t),$$

(1)

where $D = E h^3/[12(1-\nu^2)]$ is the flexural rigidity, $E$ Young’s modulus, $\nu$ Poisson’s ratio, $h$ the thickness of the plate, $\sigma$ the mass per unit area of the plate, $q$ the external transverse loading per unit area, $x$ and $y$ the spatial coordinates in the plane of the plate, and $t$ time. The boundary conditions of Eq. (1) are presented in Ref. 2, and are omitted from the presentation because of their complicated nature.

As was discussed in Ref. 3, the analytical solution of Eq. (1) for an arbitrary plate is extremely difficult if not impossible to obtain. In Ref. 3 we presented the results of a finite element analysis of the static deflection for a V-shaped cantilever (see Fig. 1); i.e., we solved

$$D\nabla^4 w(x,y) = q(x,y).$$

(2)

In this paper, we also consider the related problem of the free vibration of the plate at its natural resonant frequency. To solve this problem, we set the loading of the cantilever plate to zero, i.e., $q(x,y,t)=0$, and express the deflection function $w(x,y,t)$ as

$$w(x,y,t) = W(x,y) \cos \omega t,$$

(3)

where $\omega$ is the radial frequency of vibration and $W(x,y)$ is a function purely in terms of the spatial coordinates $x$ and $y$ (see Fig. 1). Substituting Eq. (3) into Eq. (1) we obtain

$$D\nabla^4 W(x,y) - \sigma \omega^2 W(x,y) = 0.$$  

(4)

Solving Eq. (4) under the appropriate boundary conditions will give the frequency of oscillation $\omega$ and the deflection function $W(x,y)$. As mentioned above, we are primarily concerned with the evaluation of the natural frequency of vibration and hence shall not present results for the deflection function $W(x,y)$. We must also emphasize that we are only concerned with the fundamental resonant frequency.

A. Scaling of problem

We shall now briefly discuss the scaling for the spring constant $k$ and the natural frequency of vibration $\omega$. Clearly, in order to make the results nondimensional, we must define a universal scaling length $\alpha$ for the cantilever. This choice is purely arbitrary and we choose

$$\alpha = \sqrt{A_0}.$$  

(5)

where $A_0$ is the area of the cantilever. Since the spring constant $k$ is defined to be the force per unit length deflection of the cantilever at a given point, it is evident from Eq. (2) that the appropriate scaled quantity $k$ for the spring constant is

$$\lambda_k = \frac{\alpha^2 k}{D}.$$  

(6)

Furthermore, from Eq. (4) it is also seen that the appropriate scaled quantity $\omega_0$ for the frequency of oscillation is

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**B. Normalized effective mass**

We have thus established that \( \lambda_k \) and \( \omega \) are length scale invariant quantities for the spring constant and resonant frequency of the cantilever, respectively, i.e., they depend on the scaled dimensions of the cantilever, and not the length scale of the plate. We also note that Young's modulus \( E \) appears explicitly in both \( \lambda_k \) and \( \omega \), through the flexural rigidity \( D \), and can be completely eliminated by defining a normalized effective mass \( M_e \) to be

\[ M_e = \frac{\lambda_k}{\omega} = \frac{k}{m \omega^2}, \] (8)

where \( m \) is the mass of the cantilever. Thus we have shown that the normalized effective mass \( M_e \) is a length scale invariant quantity of the cantilever. \( M_e \) is the primary function under consideration in the present method and relates the resonant frequency of the cantilever to its spring constant. Thus if the mass, dimensions, and resonant frequency of the cantilever are known, then the spring constant can be determined directly from Eq. (8).

We have solved Eq. (2) (as in Ref. 3) and Eq. (4) separately for the V-shaped cantilever, via a finite element analysis\(^3\) assuming that the load point is at the end tip of the cantilever (see Fig. 1) in the calculation of the spring constant from Eq. (2). In doing so, we have obtained results for \( M_e \) for various aspect ratios \( A = b/L \), width ratio \( d/b \), and Poisson's ratio \( \nu \), the results of which are presented in Figs. 2(a)–2(c). We also present results for a rectangular cantilever of varying aspect ratio in Fig. 3. Note that for aspect ratios less than 0.2, corresponding to \( L/b > 5 \) (see Fig. 3), as is currently encountered in practice, the value of \( M_e = 0.2427 \) may be taken. The results presented in Figs. 2 and 3 encompass practical AFM cantilevers in current use.

We emphasize that in practice care must be exercised in using the above results and method, in order to obtain accurate results for the spring constant, as we shall now discuss in detail.

**C. Off-end loading**

We note that all calculations presented have been made with the assumption of a point load at the center of the end tip of the cantilever (see Fig. 1). However, in practice the load may be applied away from the end point. This can result in large discrepancies in the spring constants found from the graphs in Figs. 2 and 3.

We first consider the case of a rectangular cantilever. In practice the aspect ratios (defined to be the ratio of the width to the length) of these cantilevers are less than 0.2 and hence it is clear that the approximate analysis presented in Ref. 3 is applicable and there is no need to resort to a full numerical analysis of the plate. From the simplified theory in Ref. 3 it can easily be shown that the spring constant \( k \), evaluated when the load is applied a distance \( \Delta L \) away from the end tip, is related to the end tip spring constant \( k_E \) by

\[ k = k_E \left( \frac{L}{L-\Delta L} \right)^3, \] (9)

where \( L \) is the length of the cantilever. Note that this relationship is accurate if the load is symmetric, since the results presented in Fig. 3 are for symmetric loads. However, we also found via a finite element analysis\(^3\) that if the load was applied off-axis this only resulted in a deviation in the spring constant of at most 2% for aspect ratios less than 0.2.

We now turn our attention to the case of the V-shaped cantilever. In Figs. 4(a)–4(c) we present results for the off-axis loading of a V-shaped cantilever of varying aspect ratio \( A \), width ratio \( d/b \), and Poisson's ratio \( \nu \), for \( k/k_E \), obtained

\[ \lambda = \omega 2 \sqrt{\frac{\sigma}{D}} \]. (7)

via a rigorous finite element analysis\textsuperscript{13} of the cantilever. Again note that a small lengthwise deviation in the load point can result in a large deviation in the spring constant. Also, we note that the results presented in Figs. 4(a)--4(c) are only for on-axis loading. If the load was applied off-axis, we found that the resulting spring constant only deviated from the on-axis value by a very small amount; e.g., for $A=1$, $d/b=0.2$, $v=0.25$, $\Delta L/L=0.2$, the maximum deviation in the off-axis load from the on-axis value was only $\sim 1\%$. This is of course due to the excellent lateral stability of the V-shaped cantilever. We also note that Neumeister \textit{et al.}\textsuperscript{4} recently presented an analytical study of the off-end loading of a V-shaped cantilever. However, they only presented numerical results for one set of geometric and material parameters of the V-shaped cantilever. In Figs. 4(a)--4(c) we present results for a wide range of geometric and material parameters, which we believe will be of considerable practical value to the users of the AFM, as we shall discuss.

**D. Air damping**

We now turn our attention to the effect of air damping on the measurements. We emphasize that all the above theoretical results have been obtained under the assumption that the cantilevers are in vacuum. Placing the cantilevers in air will clearly dampen their vibrations, leading to a shift in their resonant frequencies. As has been shown previously\textsuperscript{5}, air damping can produce significant shifts in the resonant frequencies, $\sim 5\%$ for AFM cantilevers. This is significant in the present method since an error of this magnitude will result in errors of the order of $10\%$ in the determination of the spring constant.

Clearly, if we are to account for this air damping we must examine the hydrodynamics of the flow. To this end, we examine two important parameters of the flow: the Reynolds number $\text{Re}$ and the boundary layer thickness $\delta$, which are respectively defined\textsuperscript{14} as

\begin{equation}
\text{Re} = \frac{\rho \omega X^2}{\eta},
\end{equation}

\begin{equation}
\delta = \sqrt{\frac{2 \eta}{\omega \rho}},
\end{equation}

where $\omega$ is the radial frequency of vibration, $X$ the characteristic scaling length over which the velocity varies, $\eta$ the viscosity of air, and $\rho$ the density of air. At this stage we note that Chen \textit{et al.}\textsuperscript{15} recently examined the effects of gas damping on AFM cantilevers; however, they made no comparison between the resonant frequencies in air and in vacuum, which is the present case under consideration. Furthermore, there appears to be a contradiction in the definitions of the Reynolds numbers presented in Eq. (10a) and that of Chen \textit{et al.}\textsuperscript{15} which we shall now clarify. Chen \textit{et al.}\textsuperscript{15} defined the Reynolds number to be $\rho \omega X \beta / \eta$, where $\beta$ is the amplitude of oscillation, and in so doing came to the conclusion that the
oscillations are in the low Reynolds number regime. However, the Reynolds number defined by Chen et al. only indicates the relative importance of the nonlinear inertia term to the viscous term in the Navier-Stokes equation and gives no insight into the relative importance of the linear inertia term. For small amplitude oscillations, however, the linear inertia term dominates the nonlinear term, and the Reynolds number must therefore be derived using the linear inertia term, as is the case in obtaining Eq. (10a). Therefore, if we take typical values such as $\omega \sim 2 \times 10^5 \text{ rad s}^{-1}, \nu \sim 20 \mu m$, $\eta/\rho \sim 1.5 \times 10^{-5} \text{ m}^2 \text{s}^{-1}$, we find that $Re \sim 5$ and $\delta \sim 10 \mu m$. We are clearly in the unfavorable regime where both inertial and viscous effects are important in the flow and thus any analysis which is valid for $Re \ll 1$ or $Re \gg 1$ is, strictly speaking, inapplicable.

Even so, we shall examine the accuracies which can be obtained from these limiting forms for the case of a rectangular cantilever. The analysis of the flow around a V-shaped cantilever presents a formidable challenge and shall not be attempted in this paper. Therefore we choose the rectangular cantilever as our test case, because its results for $Re \ll 1$ and $Re \gg 1$ are well known. The first case considered is that corresponding to $Re \ll 1$, where it is clear that the damping due to the surrounding air is proportional to the velocity of the cantilever. From this fact, it can be shown, in an analogous manner to that of a simple harmonic oscillator, that the resonant frequency in air, $\omega_d$, is directly related to that in a vacuum, $\omega_0$, by

$$\frac{\omega_d}{\omega_0} = \sqrt{1 - \frac{1}{4Q^2}}, \quad (11)$$

where $Q$ is the quality factor of the cantilever.

The second case under consideration corresponds to $Re \gg 1$. Clearly, in this case the damping will be proportional to the acceleration of the cantilever, and the analysis of Ref. 16 is therefore expected to be valid, from which we obtain the approximate result

$$\frac{\omega_d}{\omega_0} = \sqrt{1 + \left(\frac{b \rho_a \rho_c}{4 \rho_c h}\right)}, \quad (12)$$

where $\rho_a$ and $\rho_c$ are the densities of the air and cantilever, respectively, whereas $b$ and $h$ are the width and thickness of the cantilever, respectively.

A comparison of the above simple results with experimental results shall be given in Sec. III B to examine their validity.

E. Effect of gold coating

We shall now consider the very important case of a cantilever which is coated with a layer of gold. At close to normal incidence, the reflectivity of Si$_3$N$_4$ is about 13%, corresponding to a refractive index of 2.15. To improve the signal from the cantilevers, they are often sputter coated with a film of gold. It must be emphasized that even a thin coating of gold can dramatically change the spring constant obtained from the present method, if proper consideration is not given to its effects. By adding a layer of gold, the natural resonant frequency of the cantilever may vary. This is easily seen if one considers the gold to simply act as an added mass to the cantilever; a heavier cantilever will obviously result in a lower natural resonant frequency. Thus care must be exercised in using the present method for the determination of the spring constant, since in practice many AFM cantilevers are coated with a gold layer of substantial thickness. We shall now examine how this gold layer can be accounted for theoretically.

Since the gold coating and the cantilever substrate (i.e., the uncoated cantilever) can both be considered to be isotropic materials, it is clear that the elastic properties of the composite cantilever (i.e., which includes both the gold coating and the cantilever substrate) can be accurately modeled by an effective flexural rigidity $D_e$ and effective Poisson’s ratio $\nu_e$. Then in an analogous manner to Ref. 18, the potential energy of the plate may be immediately determined, and will be a function of the effective elastic constants and the entire mass of the cantilever. The resulting plate equation can be shown to be identical to Eq. (1), when the substitutions $D \rightarrow D_e$, $\nu \rightarrow \nu_e$, and $\sigma \rightarrow \sigma_e$ are made, where $\sigma_e$ is the mass per unit area of the entire cantilever and is defined

$$\sigma_e = \rho_{\text{Cant}} h_{\text{Cant}} + \rho_{\text{Au}} h_{\text{Au}}, \quad (13)$$

where $\rho_{\text{Au}}$ is the mass density of gold, $\rho_{\text{Cant}}$ is the mass density of the cantilever substrate, $h_{\text{Au}}$ is the thickness of gold, and $h_{\text{Cant}}$ is the thickness of the cantilever substrate. Therefore, the results presented in Figs. 2 and 3 are applicable in this case, provided that Poisson’s ratio now takes on its effective value $\nu_e$, and the mass of the cantilever is taken to be its total mass. At this stage, it should be noted that the normalized effective mass $M_e$ is not an independent function of Poisson’s ratio; however, this dependence is extremely weak, as is clear from Figs. 2 and 3. Therefore, provided that the gold coating thickness is small in comparison to that of the cantilever substrate, then $\nu_e$ may be very well approximated by Poisson’s ratio of the cantilever substrate. Furthermore, even if the thickness of the gold coating is comparable to that of the cantilever substrate, this approximation to $\nu_e$ will still yield very accurate results via the present method, due to the extremely weak dependence of $M_e$ on Poisson’s ratio.

For completeness, we shall now consider some theoretical approximations which account for the gold coating. First, we consider the case in which the thickness of the gold coating is far less than that of the cantilever substrate. In this case, the gold can be considered to be an inelastic body (to zeroth order) in comparison to the cantilever substrate, whose only effect is to increase the total mass of the cantilever. Following this approach, it is clear from Eq. (8) that

$$\frac{\omega_0}{\omega_0} = \sqrt{1 + \left(\frac{\rho_{\text{Au}}/\rho_{\text{Cant}}}{h_{\text{Au}}/h_{\text{Cant}}}\right)}, \quad (14)$$

where $\omega_0$ is the frequency of the coated cantilever and $\omega_0$ is the frequency of the uncoated cantilever.

Next we present a simple approximate result, valid for all gold thicknesses, relating the spring constants of the coated and uncoated cantilevers via their elastic and geometric properties. From the above discussion and Ref. 3, it is
clear that the spring constant \( k_1 \) of the coated cantilever is approximately related to the spring constant \( k_0 \) of the uncoated cantilever by

\[
k_1 = k_0 \left( \frac{h_{Au} + h_{Cant}}{h_{Cant}} \right)^3 \frac{E_e}{E_{Cant}},
\]

where \( E_{Cant} \) is the Young’s modulus of the cantilever substrate and \( E_e \) is the effective Young’s modulus of the coated cantilever, which we approximate by

\[
E_e = \frac{E_{Au} h_{Au} + E_{Cant} h_{Cant}}{h_{Au} + h_{Cant}},
\]

where \( E_{Au} \) is the Young’s modulus of the gold coating.

III. EXPERIMENTAL RESULTS

In order to examine the validity and accuracies of the present method and the theoretical results of the preceding section, we shall now produce experimental results for each of the cases under consideration.

A. Previous method

To verify the accuracy and practicality of the present method, it is of course necessary to experimentally measure the spring constants of the cantilevers, using a previous method. A description of the experimental procedures implemented are described in the Appendix. The Cleveland method, as outlined in the Appendix, was used to verify the present method. The Cleveland method shall now be examined in detail. Using the Cleveland method, the value of \( k \) was measured for three neighboring V-shaped cantilevers from a single wafer, of which we would expect almost identical properties due to their identical dimensions and material properties, the results of which are shown in Fig. 5. Note that in Fig. 5 three straight lines are in fact shown, with two of the lines coinciding. As expected, all three cantilevers had the same unloaded resonant frequency (±0.5%). However, two of the values of \( k \) are in very good agreement, but the third is 11% higher, as is evident from Fig. 5. We believe the reason for this is the strong dependence of \( k \) on the position of the sphere (see Fig. 4). The 11% increase in \( k \) corresponds to the placement of the tungsten calibration spheres about 4% further back (i.e., about 7 \( \mu \)m). Given that the sphere diameters varied between 3 and 30 \( \mu \)m, it is unlikely that the spheres can be placed any more exactly than this. Contrary to Cleveland et al.,\(^{10}\) we also found it absolutely necessary to “glue” the spheres to the cantilevers, since with capillary “adsorption” alone, we had little control over the sphere position. The addition of a small amount of wax has a negligible effect on the calibration curve. This is because it is less than 5% of the cantilever mass, and only ~0.1% of the tungsten sphere mass, as calculated from the change in resonant frequency when the wax was added to the tip. We believe that these variations in the sphere position are the major source of error with the technique of the Cleveland method, and that in general the values of \( k \) possess an accuracy of the order of 10%, when the spheres are carefully placed.

B. Air damping

It must be noted that all calculations presented in Figs. 2 and 3 have been performed for vibrations in a vacuum. Therefore the measured values of the unloaded resonant frequencies of the cantilevers in air must be corrected for air damping before their use with Eq. (8) in the determination of the spring constants. Since the correction formulas presented in Sec. II D differ considerably, both in functional form and in assumptions used, we measured the pressure dependence of \( \omega \) for the cantilevers used. A typical curve is shown in Fig. 6, and we found that the resonant frequency increases by about 4% in vacuum for V-shaped cantilevers and about 2% for rectangular cantilevers. Since this corresponds to a 4%–8% increase in the predicted value of \( k \), the correction is clearly important, unless the measurements are performed in a vacuum. We examined the accuracies of the formulas presented in Sec. II D for the correction to the resonant frequency of rectangular cantilevers due to air damping, for both high and low Reynolds numbers. However, we found that the low Reynolds number formula, Eq. (11), only gave a shift of ~0.03% whereas the high Reynolds number formula, Eq. (12), gave a shift of ~0.2%. Clearly both theoretical results are far smaller than that measured experimentally. Furthermore, it appears unlikely that a theoretical result at moderate Reynolds numbers would account for the 2%–4% increase in the measured resonant frequency.
shift, since both Eqs. (11) and (12) gave results at least and order of magnitude less than that required. However, this would have to be examined rigorously to make a definite conclusion. Therefore, at this time we are unable to theoretically account for the dramatic effect of air damping on the resonant frequency shift. Regardless of this, the reader should note our important observation that the typical shifts in the resonant frequencies for V-shaped and rectangular cantilevers are 4% and 2%, respectively. It should also be noted that these shifts are consistent with the observations of Albrecht et al.

We also examined the importance of air damping on the resonant frequency of a loaded cantilever, as is pertinent to the Cleveland method. Unlike the unloaded case, we found a shift of only 1% in the resonant frequency due to air damping, for a V-shaped cantilever with a tungsten sphere attached to its end tip (the ratio of the mass of the sphere to that of the cantilever was 15.5). This is as expected, since air damping must clearly have a lesser effect on a heavier cantilever.

C. Comparison of spring constants

In Fig. 7, we present a schematic depiction of the four V-shaped and two rectangular AFM cantilevers under consideration in this section, which were obtained from the same wafer. A listing of their dimensions is given in Table I. In Table II we present a comparison of the results obtained by the present method and the Cleveland method. Note that in the determination of the spring constants via the Cleveland method, great care was taken to ensure that the spheres were placed as close to the end tip as possible, in order to obtain accurate results.

Young's modulus can also be obtained from the results presented in Table II, by incorporating the results presented in Ref. 3, namely Eq. (24) of Ref. 3 and Eq. (8) of the present paper, from which we obtain a value of $E = 130 \pm 5$ GPa. As is discussed in the Appendix, the measurement error in the thicknesses of the cantilevers is ±10 nm. Hence the 4% error associated with the above value for Young's modulus is clearly consistent with the 2% error in $h$, since from Eq. (24) of Ref. 3 and Eq. (8) it is clear that ($\%$ error in $E$)$=(2\times$% error in $h$). These results are also consistent with our expectation that the material properties of all cantilevers are uniform across the entire wafer. It should be noted that Drummond and Sender$^{20}$ reported very large differences in $E$ across different wafers.

D. Off-end loading: Application to colloidal probes

At present there is a large amount of research activity focused on the measurement of forces in solution between flat surfaces and colloid probes attached to AFM cantilevers. However, as we pointed out above, the restoring force of the cantilever acting on an attached sphere is a strong function of the sphere position. A glance at the numerous micrographs of colloid probes (e.g., Refs. 8 and 21–23) reveals that in many cases, these spheres are attached at least 10%–15% back from the cantilever tip. Therefore the spring constants are 35%–60% higher than that predicted by theory, which assumes that the load is at the end tip of the cantilever. In Fig. 4 we showed how the spring constant varies with position back from the tip of a V-shaped cantilever, whereas Eq. (9) describes the variation for a rectangular cantilever. In order to experimentally verify these calculations, we have used a modification of the Cleveland method. In particular we mea-

![FIG. 7. Schematic depiction of the six AFM cantilevers used in Secs. III C–III E.](image)

TABLE I. Table showing dimensions of all six cantilevers depicted in Fig. 7. For V-shaped cantilevers, $b$, $L$, and $d$ are as described in Fig. 1. For rectangular cantilevers, $b$ and $L$ are as described in Fig. 3. All cantilevers had a thickness of $h=0.64 \, \mu$m.

<table>
<thead>
<tr>
<th>Cantilever</th>
<th>$b$ ((\mu m))</th>
<th>$L$ ((\mu m))</th>
<th>$d$ ((\mu m))</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>182</td>
<td>182</td>
<td>37.4</td>
</tr>
<tr>
<td>2</td>
<td>178</td>
<td>176</td>
<td>16.4</td>
</tr>
<tr>
<td>3</td>
<td>11.9</td>
<td>101</td>
<td>...</td>
</tr>
<tr>
<td>4</td>
<td>90.3</td>
<td>89.2</td>
<td>14.1</td>
</tr>
<tr>
<td>5</td>
<td>90.4</td>
<td>89.4</td>
<td>24.2</td>
</tr>
<tr>
<td>6</td>
<td>21.7</td>
<td>101</td>
<td>...</td>
</tr>
</tbody>
</table>

TABLE II. Table showing comparison of spring constants determined by the Cleveland method, $k_{\text{cl}}$, and the present method, $k_{\text{new}}$. $f_{\text{un}}$ is the unloaded resonant frequency measured in air. $f_{\text{vac}}$ is the corrected unloaded resonant frequency in vacuum.

<table>
<thead>
<tr>
<th>Cantilever</th>
<th>$f_{\text{un}}$ (kHz)</th>
<th>$f_{\text{vac}}$ (kHz)</th>
<th>$k_{\text{cl}}$ (N m$^{-1}$)</th>
<th>$k_{\text{new}}$ (N m$^{-1}$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>28.4</td>
<td>29.5</td>
<td>0.091</td>
<td>0.092</td>
</tr>
<tr>
<td>2</td>
<td>23.7</td>
<td>24.6</td>
<td>0.044</td>
<td>0.044</td>
</tr>
<tr>
<td>3</td>
<td>65</td>
<td>66.3</td>
<td>0.092</td>
<td>0.091</td>
</tr>
<tr>
<td>4</td>
<td>105</td>
<td>109</td>
<td>0.28</td>
<td>0.29</td>
</tr>
<tr>
<td>5</td>
<td>130</td>
<td>135</td>
<td>0.46</td>
<td>0.47</td>
</tr>
<tr>
<td>6</td>
<td>65</td>
<td>66.3</td>
<td>0.16</td>
<td>0.16</td>
</tr>
</tbody>
</table>
E. Effect of gold coating

To determine whether the discussion and simple theory presented in Sec. II E satisfactorily accounts for the effect of gold coating on the resonant frequency of the cantilevers, here we experimentally examine its effects.

We first consider the case in which the gold coating thickness is far less than that of the cantilever substrate. The results for V-shaped cantilever 1 are shown in Fig. 9. As can be seen, even a 100 Å thick layer leads to a several percent decrease in the observed resonant frequency. The solid curves shown are results obtained using Eq. (14), in which the added gold is assumed to act simply as an inelastic body with no effect on the elastic properties (i.e., spring constant) of the underlying cantilever substrate. Note that the density of the gold has to be taken to have a value of $\rho_{Au}=18.88\ g\ cm^{-3}$. As can be seen, there is excellent agreement up to 600 Å, when uncertainties of 5% in the gold thickness are included, confirming that the gold coating can be accounted for by the simple theory discussed in Sec. II E. This is of course provided that the thickness of the gold coating is small in comparison to the thickness of the cantilever substrate.

It was predicted in Sec. II E that the present method will be valid even when the gold coating substantially alters the elastic properties of the entire cantilever. In Table III, we show results where the gold coating thickness has been substantially increased, so as to affect the spring constants. Note that the Cleveland method is clearly expected to work for such large gold thicknesses. With a knowledge of the thickness and hence the mass of the gold deposited, we were able to calculate the total mass of the cantilever. With the measurement of the unloaded resonant frequencies of the cantilevers, we were then able to predict their spring constants using the present method. We also present results of the implementation of the approximate expression in Eq. (15), where we have used values of $E_{Can}=130$ GPa (as calculated above) and $E_{Au}=78$ GPa (the bulk value of Au). As is clear from Table III, there is excellent agreement between the results of the present method and the Cleveland method. Note also that the approximate expression (15) also gives good agreement with both methods, thus supporting their results. These results therefore validate our discussion in Sec. II E and clearly indicate that the present method is valid for both uncoated and coated cantilevers, irrespective of the thickness of gold coating used.

IV. MASS OF CANTILEVER

Two parameters required in the present method are the resonant frequency and the mass of the cantilever. In the

<table>
<thead>
<tr>
<th>Cantilever</th>
<th>$k_0$ (N m$^{-1}$)</th>
<th>$k_{cw}$ (N m$^{-1}$)</th>
<th>$k_{lw}$ (N m$^{-1}$)</th>
<th>$k_{pp}$ (N m$^{-1}$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.092</td>
<td>0.185</td>
<td>0.172</td>
<td>0.19</td>
</tr>
<tr>
<td>2</td>
<td>0.044</td>
<td>0.103</td>
<td>0.108</td>
<td>0.090</td>
</tr>
</tbody>
</table>
preceding sections we discussed in detail the determination of the resonant frequency and factors influencing its behavior. In this section we shall examine how the mass of the cantilever can be determined.

A. Mass via density

First we shall consider the evaluation of the mass via its density, as is common practice. Clearly, if this approach is used then the thicknesses of the cantilever substrates and gold coatings must be determined and the effects of gold coating accounted for, according to the discussion presented in Sec. II E.

LPCVD silicon nitride has elastic and physical properties that depend critically on the preparative conditions. The density, refractive index, and stoichiometry all vary considerably depending on reactor conditions and a number of kinetic factors such as gas ratio and temperature. There are basically two ways the density of the cantilevers can be obtained. The first method is simply to record the mass increase of the wafer after deposition and to measure the thickness of the wafer. This can be done ellipsometrically or with a depth profiler. This method was used by Zhang et al. A more indirect method is from the refractive index and composition of the film. The Lorentz–Lorenz equation is then used to find the density. The major drawback is that the atomic polarizabilities vary considerably with film stoichiometry, so the fits obtained will still vary with the preparative method. Furthermore, accurate composition data is required via Rutherford backscattering or other surface analytical techniques. There are only a few studies on the density of silicon nitride films under conditions similar to those employed by Park. Park employs a gas pressure of 0.6 Torr, a temperature of 785 °C, and a NH₃/SiH₄Cl₂ ratio of 1:6. Their cantilevers have a refractive index of about 2.15 (Ref. 34) due to excess silicon, compared with a stoichiometric value of 2.02 at 2 eV (620 nm). Makino has examined the properties of LPCVD Si₃N₄ under almost identical conditions. He found that a refractive index of 2.15 corresponds to a N:Si ratio of 1.1. This ratio in turn corresponded to a density of 2.8–2.9 g cm⁻³. The only other work using SiH₄Cl₂ in which the refractive index is correlated with the density is that of Zhang et al. They measured the refractive index of wafers as a function of their position within the CVD reactor. There is a considerable spread in the values of the refractive indices obtained. They also observed that SiNx layers with refractive indices around 2.1–2.2 had densities of 2.7–3.1 g cm⁻³. However, we should also note that they found a number of wafers with much higher densities around 3.7 g cm⁻³. Gyulai et al. used SiH₄/NH₃ to produce their films. Their refractive indices and N:Si ratios were similar to those obtained by Makino, though their densities were distinctly lower. However, for a refractive index of 2.20 they found a density of 2.72 g cm⁻³ and for a refractive index of 2.0, a value of 2.82 g cm⁻³.

It is apparent that if the manufacturers of AFM cantilevers provided the density of the silicon nitride, measured during manufacture, the evaluation of the spring constants would be routine. Alternatively, if the refractive index and stoichiometry of the silicon nitride films were to be provided, then the densities of individual cantilevers of arbitrary shape could be determined through calibration data such as that of Makino, Gyulai et al., or Sinha. B. Direct determination of mass

Since the normalized effective mass Mₑ is defined explicitly in terms of the mass of the cantilever, then it would be extremely advantageous if this value could be determined directly, without resort to the use of the density, for reasons discussed in the preceding section. This is the topic we shall address in this section.

In Secs. II and III we established that a gold coated cantilever can be accurately modeled by a set of effective elastic properties. Furthermore, we established that the present method is valid irrespective of the thickness of the gold coating. It is clear from this important finding that all that is required in the determination of the spring constant is the resonant frequency, dimensions, and the total mass (including the gold coating, if there is any) of the cantilever, which are then substituted directly into Eq. (8). Upon consultation with a manufacturer of AFM cantilevers, it was brought to our attention that it is not customary to measure the mass of these cantilevers in production; however, the technology now existed which could make the measurement of the total mass of the cantilevers routine, if required. This would involve the weighing of the wafers before and after deposition and also before and after gold coating. Then, with a knowledge of the area of the cantilevers, their total mass could be directly determined. If this were performed routinely and the manufacturers specified the total mass of the cantilevers, it would completely eliminate the necessity for the determination of the density, which, as was discussed in the previous section, depends greatly on the manufacturing process. Furthermore, this would completely eliminate the requirement for the consideration of gold coating, the thickness of gold, and the thickness of the cantilever substrate, as discussed above, since all that would be required would be the total mass of the cantilever. Thus the determination of the spring constants of the cantilevers by the present method would be an almost trivial procedure, since the only variable which would have to be measured would be the unloaded resonant frequency. We strongly feel that this direction should be pursued by the manufacturers of these cantilevers, since the knowledge of the total mass of these cantilevers, we believe, will be invaluable to the user of the AFM.

A computer program for calculating spring constants is available from the authors, allowing spring constants to be calculated for arbitrary V-shaped cantilevers, using either Young’s modulus or the present method. It also allows for the important variation in the load position.

ACKNOWLEDGMENTS

The authors would like to express their gratitude to K. Kjoller of Digital Instruments and M. Tortone of Park Scientific Instruments for many useful discussions and for providing details on the preparation of their microcantilevers. The authors are also indebted to Dr. J. F. Williams of the Department of Mechanical Engineering for many interesting
and stimulating discussions and for providing access to the finite element package used in this paper. The authors also wish to thank Dr. F. Caruso of CSIRO for assistance with the gold coating. One of the authors (P. M.) gratefully acknowledges the receipt of a QEII Fellowship. This research was carried out with the support of the ARC Advanced Mineral Products Special Research Centre, and was also supported in part by ARC Grant No. A69230979.

APPENDIX

In this Appendix we describe the experimental procedures implemented in this paper.

All measurements were made on a Digital Instruments Nanoscope III. The cantilevers used were procured from Park Scientific Instruments and were not gold coated. The dimensions of the various cantilevers were determined by optical microscopy using a 1 μm diffraction grating as calibrant. The thicknesses of the cantilevers were determined using a scanning electron microscope to an accuracy of ±10 nm. Resonant frequency measurements were made by taking a slope equal to $\frac{k}{M^* + M_0}$, where $M^*$ is the effective cantilever mass, and $M_0$ is the end-loaded mass. A plot of the added mass $M^*$ vs $\omega^2$ for the end-loaded mass, $M_0$ (=$mM_e$) the effective cantilever mass, and $\omega$ the angular resonant frequency. A plot of the added mass $M^*$ vs $\omega^2$ has a slope equal to $k$ and an intercept equal to the effective cantilever mass $M_0$. A small amount of wax was used to attach the tungsten spheres to the cantilevers. This allowed more exact and reproducible placement of the spheres. The spheres were mounted using ultrathin copper wires on a three-way micrometer stage. We also examined the effects of gold coating on the resonant frequency. Gold was sputter coated onto the cantilevers at a deposition rate of 1.5 Å s$^{-1}$. The thicknesses were determined using a quartz-crystal microbalance.

To examine the effects of air damping on the shape and intensity of the cantilever resonance, the Nanoscope III was placed in a specially constructed bell jar, adapted so that the photodiode voltage leads and the AFM control leads could be fed in through the base. The bell jar was then evacuated on a vacuum line down to $6 \times 10^{-4}$ Torr. Readings were taken at various pressures and the position of the peak was recorded. The resonant frequency at the peak could be read to within an accuracy of 0.5%. To prevent heating of the electronic components, the AFM was switched off between readings, and while the pressure within the bell jar was being adjusted. However, even at low pressure, no problems were encountered working in vacuum.

13. PAPEC is a trademark of, and is available from, PAPEC Ltd., Strelley Hall, Main Street, Strelley, Nottingham, NG8 6PE, United Kingdom.
20. C. J. Drummond and T. J. Senden, conference submission.
33. Park Scientific Instruments, 1171 Borregas Ave., Sunnyvale, CA 94089-1304.
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