

## Surface stress effects on the resonance properties of cantilever sensors

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(Received 16 March 2005; published 1 August 2005)

The effect of surface stress on the resonance frequency of a cantilever sensor is modeled analytically by incorporating strain-dependent surface stress terms into the equations of motion. This mechanistic approach can be equated with a corresponding thermodynamic description, allowing basic equations to be derived that link the analysis to experimentally determined parameters. Examples are shown for the cases of a pure surface stress and an adsorption-induced surface stress, and indicate that frequency measurements may be useful for fundamental understanding of surface and adsorption-induced stresses on metals, semiconductors, and nanoscale structures. Application to biomolecular adsorption sensors appears unlikely.

DOI: [10.1103/PhysRevB.72.085405](https://doi.org/10.1103/PhysRevB.72.085405)

PACS number(s): 68.35.Gy, 62.40.+i, 68.37.Ps, 85.85.+j

Microfabricated cantilever structures have been demonstrated to be extremely versatile sensors<sup>1-3</sup> and have potential applications in physical, chemical, and biological sciences.<sup>1-5</sup> Adsorption on a functionalized surface of a microcantilever may induce mass, damping, and stress changes of the cantilever response. One cantilever sensor technique is to monitor changes in the cantilever resonance frequency, and this method seems best suited for use in gaseous or vacuum environments to measure adsorption-induced mass loading of the cantilever. For work in liquid environments, measurement of the static bending of a cantilever arising from surface stress was proposed as the most appropriate method for detecting biomolecular adsorption<sup>6,7,10</sup> in liquids, and this indeed appears to be the case from a practical viewpoint.<sup>4,8</sup> However, there is minimal understanding of how surface stress influences the resonance frequency of a cantilever. As we show below, this problem is of interest in the quantitative measurement of mass and stress loads found using resonant frequency sensors; as a method to measure fundamental properties of surface stress; and of increasing importance to mechanical design as structures scale toward nanometer dimensions, including the development of potential nanoscale stress sensors.

Both theoretical and experimental studies<sup>9-12</sup> have been undertaken to investigate surface stress effects in microcantilevers. In this study we give a simple analytical description of surface stress effects and show how frequency measurements may be used to evaluate surface stresses. Previous theoretical analyses on the problem of adsorption-induced surface stress changes in cantilever resonance followed the treatment given by Chen *et al.*,<sup>10</sup> in which the differential surface stress induced by adsorption is simplified to external axial forces exerted on the cantilever. In this way, the problem of a self-balanced cantilever deformation due to mismatch stress without any external forces has been replaced by a problem of bending or vibration of the cantilever under an applied force. Although a taut string model<sup>10</sup> and a beam with axial force model<sup>11,12</sup> have been suggested based on this simplification, neither approach represents the correct physical model. The effective external forces are considerably overestimated in these models because in the real situation the cantilever has a free end to allow deformation or bending

to relieve the stress. For a cantilever with a free end, changes in cantilever resonance frequency due to small changes in curvature (i.e., bending) have been shown to be negligible.<sup>13</sup>

In what follows we consider changes in mechanical resonance frequency that arise from a surface stress, not changes in the bulk elastic properties of the cantilever that may arise on adsorption, e.g., by alloying,<sup>4</sup> material changes,<sup>10</sup> or deposition<sup>14</sup> of relatively thick films to create composite beam structures. Further, we use the term surface stress to denote stress in the neighborhood of the surface of the bulk. This includes pure surface stress (i.e., of a free surface), interfacial stress, and stress within a very thin adsorbed material.

Consider a cantilever used as a sensor. The experimental quantity measured is the surface stress difference,  $\Delta\tau = \tau_u - \tau_l$ , where  $\tau_u$  and  $\tau_l$  are the surface stresses on the upper and the lower surfaces, respectively (see Fig. 1). The stress terms can be described in a full tensor form. However, for clarity and with no loss of applicability, we simplify the problem by assuming a one-dimensional system with scalar, isotropic material properties. In this case, and to first order in the strain, the surface stresses may be written,<sup>15,16</sup>

$$\tau_u = a_u + b_u \varepsilon_u, \quad \tau_l = a_l + b_l \varepsilon_l, \quad (1)$$

where  $a$  is the strain-independent surface stress,  $b$  is a constant associated with the surface strain,  $\varepsilon$  is the surface strain measured from the prestressed configuration, and the subscripts  $u$  and  $l$  always refer to the upper and lower surface, respectively. The surface stress difference can be written from Eq. (1) as

$$\Delta\tau = \Delta\tau^0 + \Delta\tau^1 \quad (2)$$

with  $\Delta\tau^0 = a_u - a_l$  and  $\Delta\tau^1 = b_u \varepsilon_u - b_l \varepsilon_l$ . The influence of the surface stress on the resonance frequency of the cantilever sensor can be investigated by considering the influences due to  $\Delta\tau^0$  and  $\Delta\tau^1$ . This approach follows from that of Gurtin *et al.*<sup>17</sup> who described a cantilever with equal stress on top and bottom surfaces<sup>15</sup> [see Fig. 2(a)]. This work has been overlooked in more recent research and it seems timely to reintroduce their idea, which we extend to include the effects

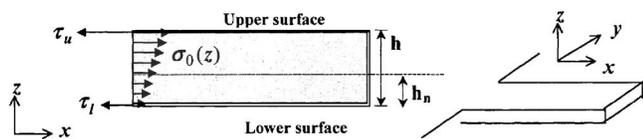


FIG. 1. Schematic view of the free-body diagram of the end part of a cantilever.

of surface adsorption and unequal stresses on top and bottom surfaces [see Fig. 2(b)].

We first consider the strain independent term,  $\Delta\tau^0$ , which can be converted to a static deflection or a static internal strain of the cantilever (similar to a temperature-induced thermal strain). If the relationship between  $\Delta\tau^0$  and the internal prestrain can be obtained, the influence of  $\Delta\tau^0$  on the dynamic properties of the cantilever sensor can be estimated using the model of a prestrained cantilever vibration.

An end-part free body diagram cut in any section of the cantilever is drawn as shown in Fig. 1. If  $a_u$  and  $a_l$  are not equal, the cantilever undergoes not only axial but also bending deformations, which is more complicated than the situation discussed by Gurtin *et al.*<sup>17</sup> The strain induced in the cantilever can be written as

$$\varepsilon_0(z) = (z - h_n)/R, \quad (3)$$

where  $R$  is the radius of curvature and  $h_n$  the distance of the neutral plane measured from the lower surface. Except for antisymmetric surface stresses, the neutral plane is generally not in the center of the sheet.<sup>18,19</sup> The bulk stress  $\sigma_0(z)$  can be expressed as

$$\sigma_0(z) = E(z - h_n)/R, \quad (4)$$

where  $E$  is Young's modulus. Note that if the system has a planar geometry,  $E$  is replaced by an isotropic biaxial modulus,  $E/(1-\nu)$ , where  $\nu$  is Poisson's ratio. The constants  $h_n$  and  $R$  can be determined from the equilibrium relations,

$$\int_0^h \sigma_0(z) dz + a_u + a_l = 0 \quad \text{and} \quad \int_0^h \sigma_0(z) z dz + a_u h = 0,$$

to give Stoney's equation,<sup>20</sup>

$$h_n = \frac{1}{Eh} R(\Delta\tau^0 + 2a_l) + \frac{h}{2}, \quad \frac{1}{R} = -\frac{6}{Eh^2} \Delta\tau^0. \quad (5)$$

The residual strain due to the surface stress is then obtained as

$$\varepsilon_0(z) = -\frac{2}{Eh} \left[ a_l + \left( \frac{3}{h} z - 1 \right) \Delta\tau^0 \right]. \quad (6)$$

Equation (6) expresses how the influence of  $\Delta\tau^0$  on the cantilever mechanical deformation has been converted to the coupling of the bending extension with the equivalent prestrain,  $\varepsilon_0(z)$ .

We now consider the cantilever vibration. The elastic stress-strain relation of the cantilever in vibration can be expressed as  $\sigma(x, z, t) = E[\varepsilon(x, z, t) - \varepsilon_0(z)]$ , where  $\varepsilon$  is the total elastic strain in the cantilever and is given by

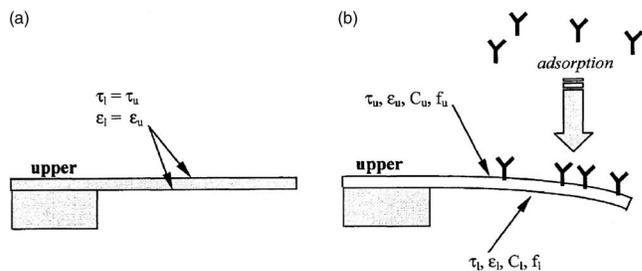


FIG. 2. Schematic showing the general cases considered for (a) a cantilever with uniform surface stress on upper and lower surfaces and no adsorption; (b) the cantilever of (a) with adsorption on one side of the cantilever, causing a surface stress difference between the upper and lower surfaces.

$$\varepsilon = -(z - h/2) \partial^2 w(x, t) / \partial x^2, \quad (7)$$

where  $w$  is the central plane deflection of the cantilever. The resultant moment  $M$  on any cross section of the cantilever is

$$M(x, t) = \int_0^h \left( z - \frac{h}{2} \right) \sigma dz = -EI \frac{\partial^2 w}{\partial x^2} + \frac{h}{2} \Delta\tau^0, \quad (8)$$

where  $I = h^3/12$  is the moment of inertia. Note that  $\Delta\tau^0$  produces a constant bending moment on the cross section. Using the resultant moment given in Eq. (8) the beam bending equation is

$$EI \partial^4 w / \partial x^4 + \rho A \partial^2 w / \partial t^2 = 0, \quad (9)$$

where  $\rho A$  is the mass per unit length. Since the equation of motion is unchanged<sup>21</sup> by  $\Delta\tau^0$ , the strain-independent part of the surface stress has no effect on the resonance frequencies of the cantilever. This is the same conclusion stated by Gurtin *et al.*<sup>17</sup> for identical upper and lower surface stresses ( $a_u = a_l$ ) but contradicts more recent modeling of the problem,<sup>10-12</sup> which leads to changed equations of motion dependant on the *constant* value ( $a_u, a_l$ ) of the surface stress.

We now consider the strain-dependent stress term,  $\Delta\tau^1$ . By replacing  $\Delta\tau^0$  in Eq. (8) with the total surface stress difference  $\Delta\tau$  defined in Eq. (2), the bending moment can be obtained as

$$M(x, t) = - \left[ 1 + \frac{3}{Eh} (b_l + b_u) \right] EI \frac{\partial^2 w}{\partial x^2} + \frac{h}{2} \Delta\tau. \quad (10)$$

Thus the effective modulus ( $E^*I$ ) to be used in the equations of motion is

$$E^*I = \left[ 1 + 3 \frac{b_l + b_u}{Eh} \right] EI. \quad (11)$$

From Eq. (10) the change in fundamental resonance frequency due to surface stress ( $\Delta\omega_{stress}$ ) can be found as

$$\frac{\omega_{stress}^2 - \omega_0^2}{\omega_0^2} = \frac{E^* - E}{E} = 3 \frac{b_l + b_u}{Eh} \approx 2 \frac{\Delta\omega_{stress}}{\omega_0}, \quad (12)$$

where  $\omega_0$  is the fundamental resonance frequency with no surface stress and  $\omega_{stress}$  ( $=\omega_0 + \Delta\omega_{stress}$ ) is the new resonance frequency with surface stresses acting. Note that the effect of surface stress depends on the size ( $h$ ) of the structure<sup>22</sup> and

$\Delta\omega_{stress}$  can be positive or negative depending on the sign of  $b$ .

Equation (12) has been left in a simple format as the parameters  $a$  and  $b$  can take various forms depending on specific models, experiments, and nomenclature. We now illustrate with some specific cases.

*Case (i): Thermodynamic expression for  $b$ .* Equation (1) is a mechanistic description of the surface stress. When cantilevers are used as sensors, a thermodynamic approach is generally more useful for which Equation (1), for the upper surface, can be equated to<sup>16</sup>

$$\tau_u^L = \left. \frac{\partial \gamma_u^L}{\partial \varepsilon_u} \right|_{\varepsilon=0} + C_u \varepsilon_u, \quad (13)$$

where  $C$  is the surface elastic constant and  $\gamma$  is the surface free energy. The superscript  $L$  indicates that the Lagrange coordinate system is used in which the surface area does not change during stretching. This definition shows most clearly the definition of the parameters  $a$  and  $b$  and is particularly useful for atomistic studies of surface stress. The deformation of the surface by a bulk strain ( $\varepsilon$ ) introduces an additional thermodynamic excess, namely the surface elastic constant.

However, in cantilever experiments, we measure the actual, strained area of surface for which the Euler coordinate system (denoted by superscript  $E$ ) needs to be used and Eq. (1) is written<sup>16</sup>

$$\begin{aligned} \tau^E &= \tau_{\varepsilon=0}^E + [C - \tau_{\varepsilon=0}^E] \varepsilon \\ &= \gamma_0 + \left. \frac{\partial \gamma^E}{\partial \varepsilon} \right|_{\varepsilon=0} + \left[ C - \gamma_0 - \left. \frac{\partial \gamma^E}{\partial \varepsilon} \right|_{\varepsilon=0} \right] \varepsilon, \end{aligned} \quad (14)$$

where  $\gamma_0$  and  $\tau_{\varepsilon=0}^E$  are the surface free energy and surface stress, respectively, for the undeformed surface. For clarity the subscripts  $u$  and  $l$  are removed.

Equations (13) and (14) describe a free surface, i.e., a “pure” surface stress. Adsorption typically generates an additional interface and a successful approach to extend the modeling in this case is to separate the free surface stress ( $F$ ) and the interface stresses ( $H, G$ ) as<sup>23,24</sup>

$$F = \gamma + \frac{\partial \gamma}{\partial \varepsilon}; \quad H = \sigma + \frac{\partial \sigma}{\partial \varepsilon}; \quad G = \sigma + \frac{\partial \sigma}{\partial e}, \quad (15)$$

where  $\sigma$  is an interfacial surface energy;  $e$  is a strain associated with stretching the adsorbed phase with respect to the substrate; and  $F$ ,  $G$ , and  $H$  are given for an undeformed surface. The stress terms  $F$  and  $H$  can be inserted into Eqs. (1), (12), and (14) to account for surface stress changes in frequency of a cantilever beam. In particular, Eq. (14) can be written

$$\tau = a + b\varepsilon = F + H + (C - F - H)\varepsilon. \quad (16)$$

We assume the interface stress term  $G$  accounting for slipping between the adsorbed film and the substrate has negligible effect on the cantilever frequency because by definition this term is separated from any strain ( $\varepsilon$ ) of the cantilever, i.e., for a frequency change to be measured the physical pa-

rameter changing must couple to the bulk strain of the cantilever.

*Case (ii): Pure surface stress.* We first consider when there is no adsorption and the top and bottom surfaces are identical, as shown in Fig. 2(a). This case represents a pure surface stress of a single material. There is no bending ( $\Delta\tau = \Delta\tau^0 = \Delta\tau^1 = 0$ ) and  $b_u = b_l$ . Equation (12) is then identical to the expression obtained by Gurtin *et al.*,<sup>17</sup> namely,

$$2 \frac{\Delta\omega_{stress}}{\omega_0} = 6 \frac{b_l}{Eh} = \frac{6}{Eh} (C - F). \quad (17)$$

To estimate the magnitude of  $\Delta\omega_{stress}$  we consider a silicon cantilever. Atomistic calculations for the Si (100) surface show that  $C \approx -11.5$  N/m,<sup>22</sup> and  $F \approx -0.5$  N/m.<sup>25</sup> Using  $E = 130$  GPa we find  $b/E \approx -1 \times 10^{-10}$ , which appears to be typical for crystalline silicon and aluminum beams.<sup>17,22</sup> Thus, for a Si cantilever of thickness  $h \sim 1$   $\mu\text{m}$ , the value of  $\Delta\omega_{stress}/\omega_0$  is  $\sim 10^{-4}$  or  $\sim 100$  ppm. This frequency shift is small but measurable. If a cantilever has  $h \sim 10$  nm (e.g., a nanowire) then  $\Delta\omega_{stress}/\omega_0 \sim 10^{-2}$ , which is a significant effect. Clearly, the extent of the stress-induced resonance frequency shift ( $\Delta\omega_{stress}$ ) is influenced by the cantilever size. Due to the large surface-bulk ratio, the effects of surface stress on the elastic modulus<sup>26–29</sup> and resonance properties of nanosized structure elements are significant.<sup>22</sup>

Conversely, the use of nanosized resonators would allow for considerable improvement in the sensitivity and usage of surface stress sensors. For example, Eq. (17) shows that it may be possible to find the surface parameter ( $C-F$ ) of single crystal surfaces by measuring frequency changes of ultrathin cantilevers of varying thickness.

*Case (iii): Adsorption.* Given the considerable difficulty in measuring absolute values of surface stress ( $\tau_u, \tau_l$ ), one invariably measures only a *change* in surface stress as the material properties of the surfaces are modified. We illustrate this by considering adsorption onto the cantilever giving a change in stress measured before adsorption [Fig. 2(a)] and after adsorption [Fig. 2(b)]. Using Equation (1), the surface stress after adsorption is

$$\tau_u = a_u + \Delta a_u + (b_u + \Delta b_u)\varepsilon_u; \quad \tau_l = a_l + \Delta a_l + (b_l + \Delta b_l)\varepsilon_l, \quad (18)$$

where  $\Delta a$  and  $\Delta b$  represent the adsorption-induced change in the stress parameters. Equation (18) assumes a linear variation of  $b$  with strain since all our considerations are for elastic deformations. The change in fundamental resonance frequency [Eq. (12)] can be expressed as

$$2 \frac{\Delta\omega_{stress}}{\omega_0} = \frac{3}{Eh} (b_u + b_l) + \frac{3}{Eh} (\Delta b_u + \Delta b_l), \quad (19)$$

with the second term showing the effect of adsorption-induced surface stress.

Interestingly, Eq. (19) shows that surface stress will change the resonance even if adsorption occurs identically on both upper and lower surfaces ( $\Delta b_u = \Delta b_l$ ), whereas no static bending would be observed [since  $\Delta\tau^0 = 0$  in Eq. (5)]. However, for most sensor applications, adsorption is constrained to occur on one surface only. Assuming adsorption

only occurs on the upper cantilever surface [see Fig. 2(b)] then the resonance frequency shift arising solely from the adsorption is

$$\left(2 \frac{\Delta\omega_{stress}}{\omega_0}\right)_{adsorption} = \frac{3\Delta b_u}{Eh} = \frac{3}{Eh}(\Delta C - \Delta F - \Delta H) \quad (20)$$

with  $\Delta C$ ,  $\Delta F$ , and  $\Delta H$  representing changes in  $C$ ,  $F$ , and  $H$  on the upper surface.

Data are not readily available for the elasticity change  $\Delta C$ . Nevertheless, we can use known values of the surface stress terms  $\Delta F$  and  $\Delta H$  to estimate  $(\Delta\omega_{stress}/\omega_0)_{adsorption}$ . For semiconductor surfaces the stress can change greatly on adsorption. For example, on a clean Si (100) surface the calculated surface stress ( $\Delta F + \Delta H$ ) changes by  $\sim 0.1$  to  $\sim 1$  N/m depending on the adsorbed species (e.g., As, Ga, Ge) and the surface microstructure.<sup>25</sup> Taking typical values for a silicon microcantilever of  $E = 130$  GPa and  $h = 1$   $\mu\text{m}$ , one finds  $(\Delta\omega_{stress}/\omega_0)_{adsorption} \sim 10$  ppm. This is a measurable shift using high quality factor cantilevers. The frequency shifts will become more pronounced with the use of ultrathin cantilevers. Indeed, there are indications that such surface stress effects have been observed in the recent data of Wang *et al.*<sup>30</sup> in which UHV clean  $\sim 50$  nm thick Si (100) and Si (111) cantilevers were exposed to molecular oxygen. The fundamental resonance frequency rapidly *increased* by approximately  $\Delta\omega/\omega_0 \sim 5000$  ppm on a low exposure (8 Langmuir) to  $\text{O}_2$ . It is highly unlikely that this large frequency shift is due to other adsorption effects because (a) an increase in mass loading would *decrease* the value of  $\Delta\omega/\omega_0$ , and (b) a change in the bulk stiffness of the cantilever by formation of an oxide could increase  $\Delta\omega/\omega_0$  on adsorption but at the low temperatures used by Wang *et al.* an  $\text{O}_2$  exposure of 8L will only result in submonolayer coverage of the silicon surface.<sup>31</sup>

One can also estimate that large stress-induced frequency shifts will occur for monolayer adsorption of metals.<sup>24,25</sup> However, the application of the resonator method for biosensors is more problematic because typical surface stress changes on biomolecular adsorption are usually  $< 0.01$  N/m.<sup>5,8</sup> The estimated frequency shifts, even for very thin cantilevers, would be  $< 1$  ppm. This level of sensitivity cannot be measured at present given that such cantilever biosensors must operate in ambient or liquid environments and this results in high viscous damping of the cantilever (quality factor  $Q \approx 1000$ ). A high  $Q$  factor is essential for measurement of surface stress using the cantilever resonator method.

*Case (iv): Comparison with other adsorption effects.* We now compare surface stress effects with other adsorption-induced frequency changes. Equation (8) is the basis of computing changes in resonance frequency due to mass loading and stiffness (i.e., effective inertia) changes arising from adsorption.<sup>2,14</sup> It is readily shown to first order that mass and stiffness changes on adsorption shift the fundamental cantilever resonance frequency ( $\omega_0$ ) by an amount  $\Delta\omega_{mass}$  given by

$$2 \frac{\Delta\omega_{mass}}{\omega_0} \approx (3E_f/E - \rho_f/\rho) \frac{h_f}{h}, \quad (21)$$

where  $h$  is thickness,  $E$  is Young's modulus, and  $\rho$  is material density, with the subscript  $f$  representing the adsorbed film.

This assumes a uniform coverage on one side of the cantilever and a simple composite beam description to model the effective cantilever stiffness.<sup>14</sup> Both conditions can be generalized.

For practical purposes the adsorbed and cantilever material properties are similar and hence  $\Delta\omega_{mass}/\omega_0 \sim h_f/h$ . For a typical cantilever  $h \sim 1$   $\mu\text{m}$  and a monolayer adsorption (i.e.,  $h_f \sim 0.1$  nm) gives  $\Delta\omega_{mass}/\omega_0 \sim 100$  ppm. We note that (a) mass loading effects are the same order of magnitude or larger than surface stress effects, and (b) the sensitivity of Eq. (21) also scales with  $1/h$ . Therefore, for semiconductor and clean metal surfaces, care will be required to distinguish between  $\Delta\omega_{stress}$  and  $\Delta\omega_{mass}$  during adsorption. In biomolecular systems, adsorption-induced stress effects will cause negligible frequency shift in comparison with mass loading.

Finally, note the difference between the Young's modulus of the adsorbed film [ $E_f$  in Eq. (21)] and the surface elasticity ( $C$ ). The elasticity  $C$  is a surface excess value, i.e., the difference between the elasticity of the surface and the bulk substrate. The modulus  $E_f$  is a bulk value for the adsorbed film. For example, if the adsorbed surface material were entirely identical to the bulk, then  $\Delta C = 0$  and surface elasticity does not change the resonance frequency. In contrast for this example  $E_f = E$ , and Eq. (21) shows that the stiffness change would lead to a frequency shift of  $\Delta\omega_{mass}/\omega_0 = h_f/h$ , which reflects the change in inertia of the cantilever beam.

To summarize, we have reinvestigated<sup>17</sup> the effects of surface stress on the fundamental resonance frequency of a cantilever sensor. Our approach differs from more recent investigations of this problem,<sup>10-12</sup> which use a strain-independent (i.e., constant) surface stress, which actually has no effect on the resonance frequency. The resonance is only influenced by the strain-dependent surface stresses, which in turn can be related to a thermodynamic description to allow a comparison to be made between the analysis and experimental data.

A major conclusion is that frequency measurements may be used to evaluate surface stresses and such data can complement static beam bending experiments. This may be useful to study surface stress of semiconductors and metals, although the experiments will be challenging as it is difficult to disentangle all of the stress terms. A particularly interesting problem will be the study of the absolute surface stress of clean, single crystal surfaces in UHV.<sup>30</sup>

If the size of the cantilever is reduced to the nanometer range, the surface stress effects on the mechanical properties become considerable, which may be detrimental or beneficial depending on the application. For example, cantilever stress sensors for adsorption typically measure a static bending of the lever but measurement of the resonant frequency shift of a nanocantilever would offer some practical benefit, such as less instrumental drift. A high quality factor is still required for applications and this appears to rule out the use of the method for biomolecular sensors requiring liquid environments.

## ACKNOWLEDGMENT

We would like to thank David Srolovitz for discussions and valuable suggestions.

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