Size-dependent effective Young's modulus of silicon nitride cantilevers

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The effective Young's modulus of silicon nitride cantilevers is determined for thicknesses in the range of 20–684 nm by measuring resonance frequencies from thermal noise spectra. A significant deviation from the bulk value is observed for cantilevers thinner than 150 nm. To explain the observations we have compared the thickness dependence of the effective Young's modulus for the first and second flexural resonance mode and measured the static curvature profiles of the cantilevers. We conclude that surface stress cannot explain the observed behavior. A surface elasticity model fits the experimental data consistently. © *2009 American Institute of Physics*.
[DOI: 10.1063/1.3152772]

Micro- and nanoelectromechanical systems are widely studied for their application in sensing and actuation devices.[1] Down-scaling of such devices improves their sensitivity however at the same time mechanical properties may start to deviate from the bulk behavior. The finite-size effects have been the subject of theoretical studies for the past years.[2–8] In experimental work on single-crystalline Si cantilevers it has been shown that the Young's modulus strongly depends on the thickness.[9] This behavior has also been observed for suspended crystalline silver nanowires.[10]

In describing the properties of nanoscale devices, the bulk Young's modulus (E) is generally replaced by the effective Young's modulus (E_{eff}) to account for size-dependent effects, including surface stress. The total surface stress (Σ) can be written as a sum of a strain-independent part (σ) and a strain-dependent part (strain ϵ), which is related to surface elasticity (C_s) $\Sigma = \sigma + C_s \epsilon$.[11–15] In this letter, we study the size-dependency of the Young's modulus in silicon nitride cantilevers when one dimension (cantilever thickness) is scaled down from 684 to 20 nm. As the SiN_x is amorphous, it is difficult to distinguish between the two contributions since parameters (e.g., C_s) are unknown and difficult to calculate. However, by comparing the experimental data for the first and second mode to theory, we show that the strain-dependent part of the total surface stress is responsible for the size-dependency.

Cantilevers are fabricated from low-pressure chemical vapor deposited (LPCVD) silicon nitride (SiN_x) on Si(100) substrates and are patterned with an electron-beam pattern generator. After resist development we use reactive ion etching in a CHF_3/O_2 (20:1) plasma to transfer the pattern to the SiN_x layer. After this step cantilevers are released using a KOH etch process (15 min etching time at 85 °C; Si etch rate about 1 μm/min), yielding facetting along the (111) planes, as shown in Fig. 1(a). This process introduces a negligible undercut, so that length corrections can be disregarded.[16] Cantilevers are fabricated with the following dimensions: lengths (L) from 8 to 100 μm, widths (w) 8, 12, or 17 μm, and thicknesses (h) ranging from 20 to 684 nm.

The thickness was measured using an ellipsometer (Leitz SP) with an accuracy of 5 nm.

Cantilevers are characterized by determining resonance frequencies from thermal noise spectra acquired by an optical deflection setup.[16] Resonances up to 5 MHz are measured with a spectrum analyzer. The estimated displacement sensitivity is 1 pm/$\sqrt{Hz}$. Cantilever dynamics can be described by the Euler–Bernoulli beam theory, and the resonance frequencies are given by (ρ is the mass density)

$$f_n = \frac{\alpha_n^2}{2\pi\sqrt{12}} \frac{h}{L^2} \sqrt{\frac{E_{eff}}{\rho}}. \tag{1}$$

The mode-dependent α_n's are 1.875 and 4.694 for $n=1$ and 2, respectively. For the first and second mode, Fig. 1(b) shows this expected relation between f_n and L^{-2} for cantilevers with a thickness of 100 nm. The ratio between the slopes of the second and first mode (6.17 ± 0.03) is in good agreement with the theoretical value [$(\alpha_2/\alpha_1)^2 = 6.23$]. This indicates that Euler–Bernoulli beam theory is applicable in our experiments, ruling out size-dependent effects according to nonlocal elastic theory for Timoshenko beams.[17] From the slope of the linear fits in Fig. 1 we have determined the effective Young's modulus $E_{eff} = 214 \pm 0.2$ GPa for the first mode and $E_{eff} = 241 \pm 0.3$ GPa for the second mode. In calculating E_{eff} we have taken $\rho = 3100$ kg m^3.[18]

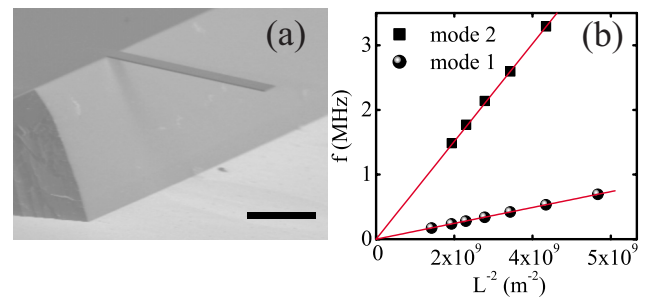FIG. 1. (Color online) (a) Scanning electron micrograph of a cantilever with dimensions $L \times w \times h = 60 \times 17 \times 0.5$ μm^3. Scale bar indicates 25 μm. (b) Resonance frequencies vs L^{-2} for the first and second mode. Cantilever dimensions: $w \times h = 12 \times 0.1$ μm^2. The drawn red lines are least square fits through the data from which E_{eff} is determined.

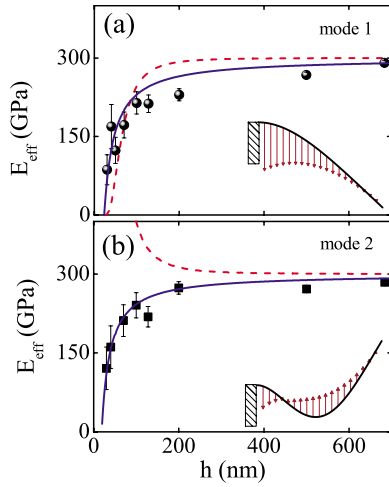a)Electronic mail: k.babaeigavan@tudelft.nl.

FIG. 2. (Color online) The effective Young's modulus vs thickness for the first (a) and second (b) flexural mode. The red dashed lines represent fits to the model incorporating strain-independent surface stress. The blue solid lines represent fits to the surface elasticity model (strain-dependent surface stress). Error bars reflect uncertainties from the least square fitting procedure. Insets show the mode shape and the corresponding transverse force.
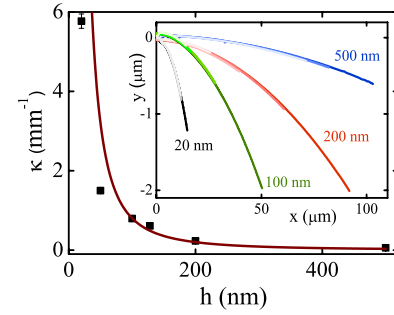

FIG. 3. (Color online) Cantilever curvature for six different thicknesses fitted to Stoney's equation (Ref. 23). The inset shows the cantilever profiles for thicknesses down to 20 nm and for different lengths. The curvature is independent of the cantilever length.

Using this procedure, the effective Young's modulus is determined for a range of thicknesses. For the first mode, the result is shown in Fig. 2(a). For $h > 150$ nm the effective Young's modulus approaches the bulk value of approximately 300 GPa.[19,18] The E_{eff} is significantly lower for cantilevers thinner than 150 nm; a reduction by a factor of three is observed for cantilevers with $h = 30$ nm.[20] A similar analysis is performed for the second mode and the results are shown in Fig. 2(b). Again, a significant decrease in E_{eff} is observed for cantilevers thinner than 150 nm, whereas for thick cantilevers E_{eff} approaches the same value as for the first mode (300 GPa). As we will show, the fact that the first and second modes show the same trend with decreasing h enables us to distinguish between surface stress and surface elasticity models. First we will discuss a recently proposed model[21] which assumes a distributed transverse force on the cantilever. A strain-independent surface stress (σ) is introduced into the Euler–Bernoulli equation to account for this force.

$$EI\frac{\partial^4 y(x,t)}{\partial x^4} - 2\sigma w\frac{\partial^2 y(x,t)}{\partial x^2} + \rho wh\frac{\partial^2 y(x,t)}{\partial t^2} = 0. \quad (2)$$

From this equation the eigenfrequencies are numerically calculated with $\sigma = 0(\omega_0)$ and with a nonzero $\sigma(\omega)$. From Eq. (1), we note that the Young's modulus is proportional to the resonance frequency squared. The effective Young's modulus is then defined by $E(\omega/\omega_0)^2$, where E is taken to be 300 GPa. We have determined E_{eff} versus thickness and the result is shown as the dashed red lines in Fig. 2. For the first mode reasonable agreement between the data and this model is found when $\sigma \approx 0.1$ Nm^{-1} [red line in Fig. 2(a)]. For the second mode the same calculation however predicts an increase in E_{eff} for thin cantilevers taking the same value of σ. The data do not show this increase in E_{eff} [Fig. 2(b)] and therefore contradict the model.

The inset in Fig. 2(a) shows the mode shape with the transverse force indicated by the arrows. For the first resonance mode, the sign of the force and displacement are the same, which can be experimentally interpreted as a decrease

in the effective Young's modulus. For the second mode [inset in Fig. 2(b)] the signs of the displacement and the transverse force are opposite. In this case the distributed force would counteract the beam displacement and cause a stiffening effect. This is in sharp contrast to our observations, and we conclude that surface stress is not the cause of the observed reduction in E_{eff}. We note that earlier theoretical work also shows that surface stress has no effect on the resonance frequency of cantilevers.[2]

In principle differential stress in the cantilevers could also affect the effective Young's modulus.[22] This stress may be built-up during the deposition process, when the composition is not homogeneous across the thickness. The resonance frequency of cantilevers is known to be dependent on such stress and the net effect may vary with cantilever thickness. Stoney's equation describes the relation between differential stress, caused by upper and lower surface layers with different stress, and the curvature $\Delta\sigma = Eh^2\kappa/3(1-\nu)$ (Ref. 23) where the curvature, $\kappa = \partial^2 y/\partial x^2$ and ν is the Poisson's ratio. To investigate the amount of residual stress in the cantilevers, the static curvature of a series of cantilevers with varying lengths and thicknesses has been measured using a white light interferometer. Figure 3 (inset) shows the measured profiles. For varying lengths but keeping the same thickness the cantilever profiles coincide, as is shown for two cantilevers with thicknesses $h = 500$ nm and $L = 70$ and 100 μm (light and dark blue lines in the inset of Fig. 3). Cantilevers with thickness down to 20 nm were measured and the curvature (κ) for each thickness was plotted in Fig. 3 (main panel). By fitting Stoney's equation to the data we find a differential surface stress of 1 Nm^{-1} where we have taken Poisson ratio $\nu = 0.24$ and $E = 300$ GPa.[24]

The differential surface stress may be the result of the SiN$_x$ LPCVD deposition: the first few nanometers may have a different composition compared to the consecutive layers due to a short stabilization time of the deposition reactor during which a low pinhole-density formation may occur.[25] Indeed microscope inspection of the cantilever base reveals a small number of pinholes in the 20 nm SiN$_x$ layer. These effects may result in a different Young's modulus for the first layers, which can be modeled by a bilayer composition.[26] We have calculated the effective Young's modulus for this case by fitting this model to the data in Fig. 2(a), and found a value of 1 GPa for the Young's modulus of the first layer with a corresponding thickness of 15 nm.[27] This low value of the Young's modulus is unrealistic given the small number of pinholes observed. Another approach to analyze the data is to

take into account the surface elasticity,[2,21] which is related to the strain-dependent part of the surface stress. Cantilevers are modeled with an infinitesimal thin surface layer (thickness $h_1 \ll h$, and Young's modulus E_1). From composite beam theory, the bending rigidity (EI) can be replaced by $EI + (1/2)C_s w h^2 + (1/6)C_s h^3$ (Ref. 21) irrespective of the mode number. Here, the surface elasticity (C_s) is defined as $E_1 h_1$, where h_1 goes to zero, but C_s stays constant. With $I = w h^3/12$ and neglecting the third term (in our case, $w \gg h$), the effective Young's modulus is written as $E_{\text{eff}} = E + 6C_s/h$. For the first resonance mode, this model is fitted to the data in Fig. 2(a) (blue line). The model, with $E = 300$ GPa as fixed parameter, is in good agreement with the data. From the fit we obtain a value of $C_s = -1170 \pm 100$ Nm^{-1}. For the second mode the model is again in good agreement with the measurements; a value of $C_s = -950 \pm 60$ Nm^{-1} is found, which is close to the value of the first mode. We note that C_s is negative for both modes, which results in a lower resonance frequency. No confirmation of our values is found for C_s in the literature for amorphous materials.

In summary, we have determined the effective Young's modulus by measuring resonance frequencies of the first and the second flexural modes of silicon nitride cantilevers with different thicknesses. For both vibrational modes the effective Young's modulus strongly decreases for thicknesses below 150 nm. The surface stress model is shown to be in contradiction with the experimental findings on the second flexural mode. The effect of differential surface stress is also found to be negligible. A surface elasticity model fits the data for both modes. Finally we note that we have also fabricated SiN$_x$ cantilevers with a dry etch technique[16] and determined E_{eff} versus h. These data show the same quantitative behavior, as in Fig. 2, indicating that the observed trend in E_{eff} is independent on the fabrication method.

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[27]A two-layer composite beam theory is used to describe the observed trend in E_{eff}. We used $E_{\text{eff}} = (k+l)/[(h+h_1)^3(Eh+E_1 h_1)]$ with $k = E^2 h^4 + E_1^2 h_1^4$ and $l = 2EE_1 hh_1(2h^2 + 2h_1^2 + 3hh_1)$. Here, h_1 and E_1 are the thickness and Young's modulus, respectively, of the first layer.