

## Characterizing size-dependent effective elastic modulus of silicon nanocantilevers using electrostatic pull-in instability

H. Sadeghian,<sup>1,2,a)</sup> C. K. Yang,<sup>2</sup> J. F. L. Goosen,<sup>1</sup> E. van der Drift,<sup>3</sup> A. Bossche,<sup>2</sup> P. J. French,<sup>2</sup> and F. van Keulen<sup>1</sup>

<sup>1</sup>Structural Optimization and Computational Mechanics (SOCM) Group, Department of Precision and Microsystems Engineering, Delft University of Technology, Mekelweg 2, Delft, Zuid Holland 2628 CD, The Netherlands

<sup>2</sup>Electronic Instrumentation Laboratory, Delft University of Technology, Mekelweg 4, Delft, Zuid Holland 2628 CD, The Netherlands

<sup>3</sup>Kavli Institute of Nanoscience, Delft University of Technology, Lorentzweg 1, Delft, Zuid Holland 2628 CJ, The Netherlands

(Received 16 April 2009; accepted 14 May 2009; published online 2 June 2009)

This letter presents the application of electrostatic pull-in instability to study the size-dependent effective Young's Modulus  $\tilde{E}$  ( $\sim 170\text{--}70$  GPa) of [110] silicon nanocantilevers (thickness  $\sim 1019\text{--}40$  nm). The presented approach shows substantial advantages over the previous methods used for characterization of nanoelectromechanical systems behaviors. The  $\tilde{E}$  is retrieved from the pull-in voltage of the structure via the electromechanical coupled equation, with a typical error of  $\leq 12\%$ , much less than previous work in the field. Measurement results show a strong size-dependence of  $\tilde{E}$ . The approach is simple and reproducible for various dimensions and can be extended to the characterization of nanobeams and nanowires. © 2009 American Institute of Physics. [DOI: 10.1063/1.3148774]

Nanocantilevers have attracted significant interest in nanoelectromechanical system (NEMS) applications.<sup>1</sup> These nanostructures are often used for actuation and sensing purposes in which the deformation and the resonance frequency highly depend on their “effective” elastic properties. Previous experimental measurements<sup>2–4</sup> and theoretical investigations (through both atomistic calculations<sup>5,6</sup> and modifications to continuum theory<sup>7,8</sup>) have indicated that the effective elastic properties of nanostructures are strongly size-dependent. Understanding and characterizing this size-dependence, in particular the effective Young's Modulus  $\tilde{E}$ , not only raises serious challenges for experimental and computational studies but also is a bottleneck to the predictability and reproducibility of performance. At small length scales, different loading methods result in different  $\tilde{E}$ .<sup>9</sup> There are in general two types of experimental characterizations: those measuring  $\tilde{E}$  in the extensional mode<sup>4</sup> and those in the bending mode.<sup>10</sup> Most of the research today focuses on the bending mode of  $\tilde{E}$  because it is more sensitive to surface stress and surface elasticity effects,<sup>4</sup> and because of its wide application in mass sensing,<sup>11</sup> force sensing,<sup>12</sup> and displacement sensing.<sup>13</sup>

When determining the stiffness of a nanocantilever, one common approach is to measure the resonant response of the system. In this case,  $\tilde{E}$  is calculated via the Euler–Bernoulli equation from fitted resonance frequencies.<sup>10,14</sup> However, the resonant response of the cantilever depends on both the stiffness and the mass. It is therefore difficult to decouple solely by resonance response, the stiffness from the mass changes caused by surface contamination, native oxide and other adsorbed layers. Consequently, the extra surface mass dominates the extra stiffness and decreases the resonance

frequency,<sup>15</sup> causing the interpreted  $\tilde{E}$  to be lower than the actual value, making the measurement qualitative and rather unreliable in high surface-to-volume ratio structures.

Another common approach is using bending test in an atomic force microscope (AFM). Such an approach has high force and displacement resolution, but it also has considerable uncertainties in its measurement and interpretations. Errors result from difficulties in: measuring the real deflection, preventing tip slippage,<sup>3</sup> determining the contact area,<sup>4</sup> and assessing the tip-cantilever indentation.<sup>4</sup> Moreover, the bending tests require an accurate measurement of the distance from the clamping point to the forcing point. This introduces a length error typical for the AFM method. Even with the latest multipoint testing protocol, the inaccuracy can sum up to more than 26%.<sup>3</sup> Finally, the probe microcantilevers are usually limited in stiffness (typically 0.01–10 N/m) and therefore cannot be used to determine appreciatively the mechanical properties of nanostructures that are either very stiff or extremely flexible.<sup>3</sup>

To avoid the aforementioned issues, we propose the use of quasistatic electrostatic pull-in instability to study the size-dependent  $\tilde{E}$ . Although a variety of experimental studies using pull-in approach at the microscale are available and it was shown to be one of the best methods for extracting the mechanical properties,<sup>16</sup> so far there is no experimental work known to us for NEMS. We show for the first time that the approach is extremely robust and accurate in measuring the  $\tilde{E}$  of ultrathin cantilevers and that it can be adapted to the measurement of other nanostructures.

The uniqueness of the pull-in method lies in its well-known sharp instability and the possibility of applying a force distributed along the length of the beam. Through the well-established electromechanical interaction, the stiffness and  $\tilde{E}$  can be accurately determined. The measurement is

<sup>a)</sup>Electronic mail: h.sadeghianmarnani@tudelft.nl.

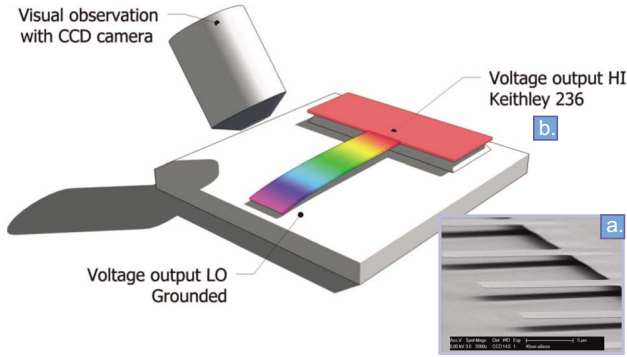


FIG. 1. (Color online) (a) SEM of 40 nm thick silicon cantilevers (the scale bar is 5  $\mu\text{m}$ ). (b) Schematic view of the measurement setup. The driving voltage on the cantilever was applied through a probe contact and the substrate was grounded.

independent of mass-loading effects and the method-induced error is the lowest among all the bending  $\tilde{E}$  measurement methods in NEMS up to this day. Furthermore, the snapping of the cantilever can be easily determined compared to AFM bending or resonance peak fitting, and a wide range of detection method can be applied. The required actuation voltage can be accurately measured using standard electrical test equipment and a microscope.<sup>16</sup>

When a driving voltage is applied between the cantilever and the substrate, the electrostatic pressure deflects the cantilever. By increasing the applied voltage beyond a critical value (called the pull-in voltage), a stable equilibrium position of the cantilever ceases to exist and the cantilever snaps toward the substrate. The pull-in behavior depends on the interaction of the electrostatic load (generated by the applied voltage), the stiffness of the cantilever and the geometry.<sup>17</sup> Through careful fabrication and precise measurement, the high stiffness sensitivity of the pull-in voltage enables a confident  $\tilde{E}$  measurement.

The general governing differential equation of the pull-in behavior is stated as<sup>16</sup>

$$\tilde{E}t^3y'''' - \frac{\alpha V^2}{y^2} = 0, \quad (1)$$

where  $t$  is the thickness of the cantilever,  $y$  is the gap between the cantilever and the substrate,  $\alpha$  is a constant, and  $V$  is the applied voltage. Due to the nonlinearity of the elastic-electrostatic interaction, exact analytical solutions are generally not available. For this, the generalized differential quadrature (GDQ) algorithm was employed to solve the nonlinear differential equation extracted from the variational calculus of energy.<sup>17</sup> By solving Eq. (1), the instability point or the pull-in voltage can be determined. Here, for each thickness, the pull-in voltage and geometry of various cantilevers with different lengths were measured. The pull-in data versus lengths of the cantilevers were fitted to Eq. (1) and solved by the GDQ algorithm. From the solution, the  $\tilde{E}$  value for each thickness was determined.

The silicon cantilevers used for investigation were all made by standard fabrication processes using silicon on insulator (SOI) wafers.<sup>18</sup> Figure 1(a) shows the scanning electron microscopy (SEM) of ultrathin cantilevers. The measurement setup consists of a voltage source (Keithley 236) and a semiconductor testing probe station; the configuration

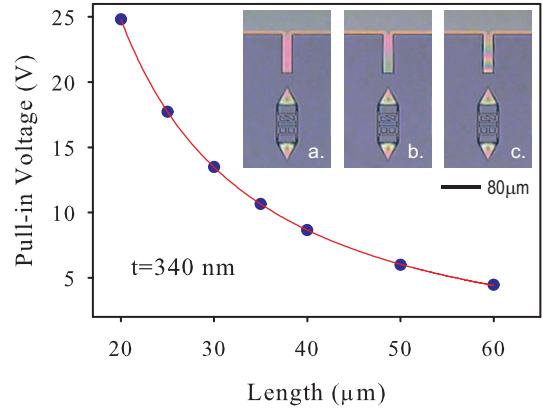


FIG. 2. (Color) A typical measurement result of an array of cantilevers with the same thickness. The pull-in points were fitted using the GDQ algorithm (solid line). Insets [(a)–(c)] show the response of a cantilever to the slowly increasing voltage up to the pull-in point (c).

is illustrated in Fig. 1(b). The pull-in voltage was measured by slowly increasing the voltage difference between the cantilever and the substrate. Visual observation of the color changes and the sudden stiction of the cantilever were used to determine the pull-in voltage.<sup>18</sup> Figure 2 shows a typical pull-in measurement on a set of cantilevers with the same thickness. Insets (a)–(c) in Fig. 2 show the top-view of a cantilever in the process of pull-in from the initial state to half way bending and finally to pull-in. Notice the substantial color change and the distinctive fringes at stiction. The measurements were repeated for thicknesses 1019, 340, 93, 57, and 40 nm.<sup>18</sup> Figure 3 shows the extracted  $\tilde{E}$  as function of the cantilever thickness. Error bars in Fig. 3 were calculated using

$$\frac{\Delta \tilde{E}}{\tilde{E}} = 4 \left| \frac{\Delta L}{L} \right| + 3 \left| \frac{\Delta t}{t} \right| + 3 \left| \frac{\Delta g}{g} \right| + 2 \left| \frac{\Delta V}{V} \right|, \quad (2)$$

where  $L$  is the length of the cantilever and  $g$  is the initial gap. Note that the first three terms are device dimensional errors, and only  $\Delta V$  is the experimental error introduced by the method. The maximum calculated error of  $\tilde{E}$  was 12%.<sup>19</sup> The main contribution comes from errors in geometry. The measured  $\tilde{E}$  of each individual cantilever (open triangles) are all

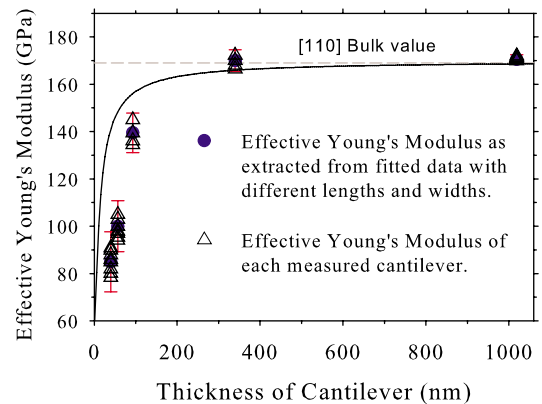


FIG. 3. (Color online) The size dependence of the  $\tilde{E}$ . The blue circles represent the  $\tilde{E}$  for an array of cantilevers, extracted from fitted results, similar to the one presented in Fig. 2. The dashed line is the bulk values for silicon [2]. The solid line is the calculated  $\tilde{E}$  through Eq. (3).

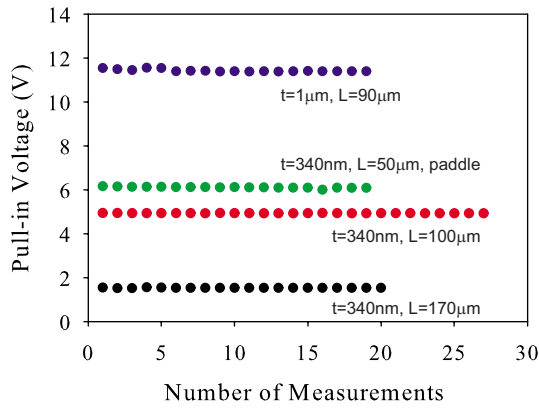


FIG. 4. (Color online) Repeated measurements on various cantilevers. The measurements have been carried out continuously.

within the error bars. The  $\tilde{E}$  is strongly size-dependent and starts to decrease monotonically somewhere near 150 nm. This decrease is consistent with theoretical predictions, in which the effects of surface elasticity<sup>8</sup>  $S$  and native oxide layers, with their distinct elastic response<sup>3,6</sup>  $E_{ox}$  are considered. Thus, the  $\tilde{E}$  can be estimated as<sup>3,8</sup>

$$\tilde{E} = E_b \left( 1 + \frac{6S}{E_b t} \right) \left\{ \frac{t^3 + \frac{E_{ox}}{E_b} [8(t_{ox})^3 + 6t^2 t_{ox} + 12t(t_{ox})^2]}{(t + 2t_{ox})^3} \right\}, \quad (3)$$

where  $E_b$  is the bulk value of Young's Modulus, and  $t_{ox}$  is the thickness of the oxide layer at top and bottom surfaces, which is about 2 nm.<sup>3</sup> The result is shown as a solid line in Fig. 3. The more pronounced experimentally observed decrease in  $\tilde{E}$  can be due to additional factors such as surface contamination<sup>6,20</sup> and crystal defects.<sup>6,10,20</sup> To address the issue of precision of the pull-in measurements, repeatability and reproducibility experiments were carried out. Figure 4 shows the results for different thicknesses, lengths, and shapes (simple and paddle), indicating that the pull-in approach is both repeatable for single cantilevers as well as reproducible for different samples.

In summary, we introduced the use of pull-in instability as an alternative approach that enables an accurate and robust measurement of  $\tilde{E}$  of nanocantilevers, as well as nanobeams and nanowires. We have shown that this approach is so far the most accurate in determining the  $\tilde{E}$  of ultrathin

cantilevers. Compared to the popular resonance response and AFM bending measurements, the pull-in approach is free from the mass-loading effect and easy to implement. However, the approach does have its limitations; the use of electrostatic force requires a fairly conductive device and a counter electrode. Moreover, stiction prevention by additional stopper designs is necessary to ensure the release of the cantilever for multiple measurements. These results are used in ongoing research into the causes of size-dependence of  $\tilde{E}$ .

This work was supported by the Dutch national research program on micro technology, MicroNed (Project code: IV-C-2), and NanoNed (Project code: TQVB46). The authors would like to thank Professor L. Nanver in helping with the equipment setups and Professor U. Staufer for discussion and valuable suggestions.

<sup>1</sup>H. G. Craighead, *Science* **290**, 1532 (2000).

<sup>2</sup>T. Kizuka, Y. Takatani, K. Asaka, and R. Yoshizaki, *Phys. Rev. B* **72**, 035333 (2005).

<sup>3</sup>M. J. Gordon, T. Baron, F. Dhalluin, P. Gentile, and P. Ferret, *Nano Lett.* **9**, 525 (2009).

<sup>4</sup>R. Agrawal, B. Peng, E. E. Gdoutos, and H. D. Espinosa, *Nano Lett.* **8**, 3668 (2008).

<sup>5</sup>B. Lee and R. E. Rudd, *Phys. Rev. B* **75**, 195328 (2007).

<sup>6</sup>M. T. McDowell, A. M. Leach, and K. Gall, *Nano Lett.* **8**, 3613 (2008).

<sup>7</sup>S. Cuenot, C. Frégnigny, S. Demoustier-Champagne, and B. Nysten, *Phys. Rev. B* **69**, 165410 (2004).

<sup>8</sup>R. E. Miller and V. B. Shenoy, *Nanotechnology* **11**, 139 (2000).

<sup>9</sup>M. T. McDowell, A. M. Leach, and K. Gall, *Modell. Simul. Mater. Sci. Eng.* **16**, 045003 (2008).

<sup>10</sup>X. Li, T. Ono, Y. Wang, and M. Esashi, *Appl. Phys. Lett.* **83**, 3081 (2003).

<sup>11</sup>Y. Yang, C. Callegari, X. Feng, K. Ekinici, and M. Roukes, *Nano Lett.* **6**, 583 (2006).

<sup>12</sup>H. J. Mamin and D. Rugar, *Appl. Phys. Lett.* **79**, 3358 (2001).

<sup>13</sup>C. Stampfer, A. Jungen, R. Linderman, D. Oberfell, S. Roth, and C. Hierold, *Nano Lett.* **6**, 1449 (2006).

<sup>14</sup>A. W. McFarland, M. A. Poggi, L. A. Bottomley, and J. S. Colton, *J. Micromech. Microeng.* **15**, 785 (2005).

<sup>15</sup>H. G. Craighead, *Nat. Nanotechnol.* **2**, 18 (2007).

<sup>16</sup>P. Osterberg and S. Senturia, *J. Microelectromech. Syst.* **6**, 107 (1997).

<sup>17</sup>H. Sadeghian, G. Rezazadeh, and P. M. Osterberg, *J. Microelectromech. Syst.* **16**, 1334 (2007).

<sup>18</sup>See EPAPS supplementary material at <http://dx.doi.org/10.1063/1.3148774> for descriptions of fabrication, alternative detection method, and full experimental measurements.

<sup>19</sup> $\Delta L$  is the length error measured by SEM.  $\Delta t$  and  $\Delta g$  are the errors of the ellipsometry measurements, and  $\Delta V$  is the accuracy of the voltage source.

<sup>20</sup>H. S. Park, W. Cai, H. D. Espinosa, and H. Huang, *MRS Bull.* **34**, 178 (2009).