Study of buckling behavior at the nanoscale through capillary adhesion force

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ABSTRACT: This paper presents mechanical actuation experiments performed on ultrathin suspended nanoscale silicon devices presenting Euler buckling. The devices are fabricated by a combination of focused ion beam (FIB) implantation and selective wet etching. By loading the center of curved nanobeams with an atomic force microscope (AFM) tip the beams can be switched from an up-buckled position to the opposite down-buckled configuration. It is possible to describe the entire snap-through process thanks to the presence of strong capillary forces that act as physical constraint between the tip and the device. The experiments conducted recall the same behavior of macro and microscale devices with similar geometry. Curved nanobeams present a bistable behavior, i.e. they are stable in both configurations, up or down-buckled. In addition to that, by the method presented, it is possible to observe the dynamic of a mechanical switch at the nanoscale.

By artificially creating compressive buckling it is possible to turn planar structures, patterned with conventional micro/nano fabrication methods, into 3D structures with different shapes and mechanical properties. One of the most interesting property of micro/nanoelectromechanical systems (MEMS/NEMS) is the so called mechanically bistable mechanism. The simplest example is represented by a single beam, clamped on both sides, that buckles under axial compression; the resulting curved beam can switch between the two stable configurations. Using standard fabrication methods, microscale beams can be fabricated and artificially buckled thanks to the introduction of surface stress, producing the very same effect of an axial compression\textsuperscript{1}. Snap-

action bistable mechanism is useful for the realization of micro-switches, microbalances\(^2\), non-volatile memories\(^3\)\(^4\) and sensors\(^5\), with the obvious advantage of not requiring power to keep the mechanism in one of the configurations. Usually, the switching between states is done statically by electrostatic actuation\(^4\)\(^6\)\(^7\) or, in alternative, driving the device into resonance\(^8\). As we scale down to nano-structures some interesting phenomena arise. For example, in ambient condition, where water is ubiquitous, capillary forces become predominant over surface forces. Adhesion forces become of the same order of magnitude of the elastic response of nano-structures and we can thus design experiments otherwise not feasible.\(^9\) In order to study bistability, it is necessary to create a dedicated set-up with separated force sensors\(^10\) and to reconstruct the actuation curves merging partial branches\(^11\). In this work we were able to study the whole dynamic of the buckling response at the nanoscale tanks to the water bridge produced by capillary condensation binding the nanobeam to the tip of an AFM cantilever.

To obtain suspended Si doubly clamped nanobeams we employed a prototyping method derived from a combination of focused ion beam (FIB) exposure, inducing local gallium implantation, and selective silicon etching, followed by high temperature annealing. The method is reported in the supplementary information (SI) and described in details elsewhere\(^12\)\(^-\)\(^15\). For the purpose of our study we fabricated three different beam geometries (type A, B and C); varying length (\(l\)) and width (\(w\)), while beams thickness (\(t\)) is kept constant, defined during the ion beam implantation by the penetration range of the gallium ions in Si. At the end of the process, \(t\) typically resulted between 30 and 40 nm. Schematic of a rectangular doubly clamped beam is reported in figure 1a. As we released the underlying Si by wet etching, the suspended beams present a certain curvature. Prior the actuation the device is imaged with AFM at low force set point, in order to measure the curvature and the distance with respect to the horizontal plane (\(h_0\)) and also to define the mid-point. Force-distance (F-d) curves have been acquired both in contact mode and in dynamic mode (the latter mode used in humidity dependent experiments). X-Y scanning is operated in closed-loop so that tip positioning in the mid-point after imaging has nm accuracy. The characteristics of the beams are summarized in TABLE I, the complete characterization is available in SI. The experiments were carried out in a closed chamber with
controlled relative humidity (RH). Cantilevers employed were commercial Si cantilevers (Bruker RTESPA 150) with nominal spring constant of 5 N/m and nominal tip radius \( R_t = 8 \) nm, selection criteria are explained in SI. Each cantilever was calibrated by the thermal tune method\(^{16}\) prior to measurements. First the sensitivity of the optical lever is measured on a sapphire plate, then thermal noise is acquired. For the relatively stiff cantilever employed (5 N/m) the combined relative uncertainty in the determination of \( k \) is 20\% (see TABLE I). We compared the data acquired by AFM with existing literature describing elastic beams with the same geometry but much larger size, ranging from macroscale\(^{10,17,18}\) to microscale Si beams\(^{11}\). We tested 6 nano-beams of different geometries, (type A, B, and C); the measurements consisting of actuation experiments performed at the beams’ axial center point as depicted in figure 1a. Displacement \((d)\) is defined as the vertical displacement of the beam-tip contact point, obtained subtracting the cantilever deflection from the z-piezo displacement; \( d \) can be alternatively defined as the beam’s center deflection.

Since beam’s surface and tip are in close contact at the beginning of actuation (side view shown in figure 1a) a water meniscus is formed due to capillary condensation. We can assume that the silicon surface implanted is slightly hydrophilic, accepting the value of water contact angle \( \theta = 31^\circ \) reported in literature\(^{19}\). A sketch of the water bridge formed is given in figure 1b, where \( R_1 \) and \( R_2 \) are the meniscus curvatures related to the Kelvin radius \( R_k \) by the Kelvin equation:

\[
\frac{1}{R_k} = \frac{1}{R_1} + \frac{1}{R_2} = \frac{RT \ln(RH)}{\gamma V_m}
\]

(1)

where \( V_m \) is the molar volume of water, \( RT \) is the temperature times the gas constant, \( \gamma \) is the liquid–vapor water surface tension and RH the relative humidity. From (1), for water vapor in air at room temperature \( R_t \) [nm] = 0.54/\( \ln(RH) \)\(^{20}\) at 40\% RH, \( R_t \approx 1.7 \) nm, compatible with a value of \( R_2 \) of 5 - 6 nm, as assumed in our experiment. The adhesion pull-off force recorded when we separate the tip from the flat surface is given by the sum of capillary forces (Laplace force and
line tension force) and other weak forces (e.g. van der Waals). For a sphere-on-plane geometry, an approximation of capillary force value is given by:

\[ F_{cap} = 4\pi R_t \gamma \cos \theta \]  

(2)

where \( R_t \) is the tip radius. Eq. (2) is humidity independent and gives a value of \( F_{cap} = 6.2 \) nN for \( R_t = 8 \) nm and \( \theta = 31^\circ \), slightly inferior to our experimental values, and is considered not fully reliable describing AFM experiments. More accurate calculations, taking into account RH, meniscus shape and pressure inside the capillary bridge, showed how capillary force is the main component at high RH but still plays an important role at RH below 20%. Several AFM pull-off experiments show that even at very low RH, adhesion force does not drop consistently, either because capillary forces do not vanish or because the van der Waals force becomes dominant. The experimental values of pull-off forces measured outside the suspended area at high (40%) and low (4%) humidity were 20 and 10 nN respectively (figure S4), comparable to other experimental results for Si AFM tip and Si samples.

An example of a mid-span actuation experiment starting from up-buckled position is given in figure 2a; the curve obtained presents the typical Z-shaped behavior similar to the mid span actuation of macroscopic doubly clamped beams, the black branch is the approach curve while the red branch is the retract, actuation speed (z piezo velocity) is 0.5 \( \mu \)m s\(^{-1}\). It is possible to identify three main branches during actuation (figure 2a), (1) the tip comes into contact and the water meniscus is formed. This is followed by a deformation of the beam until a critical load, \( F_{crit} \), is reached at \( d_{max} \). At this point a branch with negative stiffness begins (2); this is due to the spontaneous evolution of the system that passes through a higher buckling mode to finally complete the snap-through, releasing the elastic energy stored. The presence of the water bridge allows for the detection of the negative force, creating an elastic constraint between the cantilever and the beam that is able to pull down the cantilever (or pull up the beam during retract). Once the down buckled configuration is reached at \( d_{min} \), the quasi linear elastic bending (i.e. three point bending) of the beam begins (3) until the force reaches the trigger point and cantilever retracts. The adhesion force at the end of the retract curve (point 1) is close to the value of the minimum
negative force measured during actuation ($F_{\text{min}}$) explaining how the meniscus can hold the beam against the tip during the whole snap-through. The (stiffer) cantilever dominates the dynamic of the snap-trough, what observed are the static configurations experienced by the beam during the whole snap-through. What the F-d curve displays between point 1 and point 3 is the force exerted by the beam on the cantilever during the snap-through. It is possible to identity certain parameters for each curve, as shown in figure 2b. The distance between the two contact points is $2h_0$ as expected, $F_{\text{crit}}$ and $F_{\text{min}}$ represent the maximum force needed to snap the beam (for an ideally bistable symmetric beam the curve should be symmetric as well and $F_{\text{crit}} = F_{\text{min}}$). $F_{\text{crit}}$ can be calculated from the expression given by Vangbo et al.:

$$F_{\text{crit}} = -129.5 E l l^3 \sqrt{\Delta d l}$$  \hspace{1cm} (3)$$

where $l$ is the suspended length, $E = 68$ GPa is Si Young modulus for ultrathin cantilevers, $I$ is the area momentum of inertia ($I = wt^3 / 12$) and $\Delta d$ is the difference of length between the suspended length $l$ and the curved length. Experimental values and the values obtained from eq. (3) are compared in TABLE I, resulting in experimental forces slightly below the theoretical values. This might be due to two factors, an overestimation of beam thickness or an overestimation of the material Young’s modulus. If beams show a good symmetry between the up buckled and the down buckled configuration ($h_0$ is the same in both down and up buckled configuration) approach and retract overlap entirely. Most beams present a slight mismatch of symmetry, with one of the two positions being preferential. In the case of A2 (figure 2c), the down-buckled configuration is preferential, producing the visible displacement of $d_{\text{mid}}$ towards left; moreover, for all beams with asymmetry, we observe $F_{\text{crit}} < F_{\text{min}}$. For beam A1, the mismatch is almost negligible ($\approx 7$ nm), producing a symmetric z-shaped approach curve (figure 3b). Approach curves for all beams tested are presented in figure 2c-d, starting with a comparison of beams A1-3 and continuing with example actuation curves of beam B1 and C1, both “softer”, showing a decrease in $F_{\text{crit}}$. Experiments are well reproducible; each beam could stand more than 15 cycles of actuation resulting in substantially identical curves, mechanical response is not affected by repeated actuations (figure S5). The actuation can be reversed at any time, even before
completing the snap-through, showing that meniscus is acting as a constraint, as is shown in figure 3a. Tip is retracted before completing the snap through, triggered at 60 and 150 nm displacement respectively.

Qiu and coworkers\textsuperscript{11} gave a complete description of the mid-span actuation of a buckled beam only depending on a single geometric parameter, $Q = h_0/t$; the normalized applied force $F^*$ and normalized displacement $\Delta$, are defined as follows:

$$F^* = Fl^3/EIh_0, \quad \Delta = d/h_0$$ \hspace{1cm} (4)

The first three buckling modes for doubly clamped beam are schematized in figure 3b. For each of these modes there is a solution ($F^* - \Delta$ relationship) that describes the mid-span actuation, $F^*_1$, $F^*_2$ and $F^*_3$ can be written as function of $\Delta$ and $Q$:

$$F^*_1 = \frac{3\pi^4Q^2}{2} \Delta \left( \Delta - \frac{3}{2} + \frac{1}{\sqrt{4 - \frac{4}{5Q^2}}} \right) \left( \Delta - \frac{3}{2} - \frac{1}{\sqrt{4 - \frac{4}{5Q^2}}} \right)$$ \hspace{1cm} (5)

$$F^*_2 = 4.18\pi^4 - 2.18\pi^4\Delta$$ \hspace{1cm} (6)

$$F^*_3 = 8\pi^4 - 6\pi^4\Delta$$ \hspace{1cm} (7)

Those are all valid solutions and the system is likely to switch from one to the other during the actuation. “A” type beams fabricated have $Q = 1.6 – 2$; values that would not allow bistability. Since bistability is observed, we have either underestimated $h_0$ or, overestimated thickness $t$. AFM measurements of vertical position are usually accurate so it is likely that the major source of uncertainty is given by the overestimation of thickness $t$. SEM inspections revealed that the beams get thinner closer to the center (Figure S3). Moreover the edges of the cantilever are rounded; both factors could modify the value of $Q$. It is interesting to remark that $F_2$ and $F_3$ are straight lines with negative slope independent from $Q$, while only $F_1$ varies with $Q$, representing the initial and final branch of the $F$-$d$ curves. The comparison between the normalized data of beam A1 and the analytical relationship given by Qiu, is shown in figure 3c-d, for $Q = 2.5$ and $Q = 3$, assuming thinner beams; $h_0 = 25$ and 21 nm respectively. Under this assumption, experimental data overlap with $F_1$ in the first part, and then follow a straight line with negative slope equal to the slope of $F_3$. Since $F_3$ exists only with the second mode constrained, we can deduce that the meniscus binding suppress the second buckling mode. The negative branch ($F^*<0$) of the experimental
curve does not behave as predicted, suggesting that either a mismatch in symmetry and/or the presence of the meniscus generates a deviation from $F_1$. Similar results are obtained comparing the analytical relationship with the experimental data in case of beams B1 and C1 (figure S6).

Additionally to center load, it is possible to map the deformation along the cantilever horizontal axis. That means mapping the apparent stiffness during a single scan. Peak Force tapping is able to acquire a large number of F-d curves and elaborate them in real time\textsuperscript{28}, experimental details of the technique are given elsewhere\textsuperscript{29,30}. At constant maximum load, neglecting the penetration of the tip into the beam, getting a larger deformation means that the whole suspended structure bends more easily if sollicitated in the point probed (sketch in figure 3e). The deformation map collected during a scan acquired at a force set point of 1 nN is shown in figure 3e. The two peaks pink colored areas in figure 3e represent the two areas where, if we apply a normal force, we obtain actuation with the minimum load. For clarity we report further details in SI.

Another indication of bistability is given by the fact that by lowering humidity it was possible to control the switch between the up buckled and the down buckled configuration. For RH $\leq$20% we start observing a detachment during the mid-span actuation as shown in figure 4a. To investigate the meniscus role during actuation the second set of F-d curves have been acquired in dynamic mode (figure 4). During the dynamic mode approach curves at 8% RH on beam B1 we acquired both cantilever oscillation amplitude and averaged TM deflection (exerted force during tapping), amplitude feedback is turned off. The detachment between the tip and the beam is clearly visible in both graphs. The amplitude signal drops (free amplitude value $\approx 26$ nm) in coincidence with the jump to contact and is restored as the contact is lost. As expected, contact loss, produces an immediate upward jump of the cantilever, with zero force detection until the tip lowers enough to jump to contact a second time. Once triggered, the snap-through continues, as the beam ends in the down-buckled shape. The evolution of the actuation at low humidity, including approach and retract curves, with the rupture of the meniscus is schematized in figure 4b. As shown, the evolution of the actuation ends with the effective switch of the beam. Actuation experiments were conducted at 0.5 $\mu$m s$^{-1}$; with same approaching and retract speed along z. In order to test the
stability of the water neck we increased the actuation speed, starting from 100 nm s\(^{-1}\) (the quasi-static case) up to 40 µm s\(^{-1}\). For speed below 15 µm s\(^{-1}\) the experiments are reproducible and the water neck is stable (results commented in SI).

It has been shown how AFM force spectroscopy can be used under ambient condition to mechanically test suspended nanobeams. We proved that the water meniscus created acts as an elastic bound between the actuating tip and the beam. This situation allowed us to monitor the process of actuation of the doubly clamped buckled nanobeams. We were able to measure the critical load needed to trigger the snap-through and confirm the existence of a bistable behavior. Beam’s thickness represents the most sensitive parameter; small errors in the measurement of thickness generate substantial deviations from available models. The presence of the meniscus is probably capable of constraining the second buckling mode of the system. The method described here will provide useful information to quantify the adhesives forces in nanoelectromechanical switches \(^{31}\) where the interplay between elastic forces of the active elements and attractive forces as adhesion can preclude a proper device operation.

SUPPLEMENTARY MATERIAL

Supplementary information report further details regarding the fabrication and characterization of suspended beams, cantilever selection, adhesion force on Si implanted surface, Peak force characterization and F-d curves at increasing approach speed.

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Table 1. Characteristics of the three nanobeams fabricated

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<thead>
<tr>
<th>Beam</th>
<th>$w$ [nm]</th>
<th>$h_0$ [nm]</th>
<th>$l$ [$\mu$m]</th>
<th>$t$ [nm]</th>
<th>$F_{\text{crit}}$ EXP. [nN]</th>
<th>$F_{\text{min}}$ EXP. [nN]</th>
<th>$F_{\text{crit}}^*$ [nN]</th>
</tr>
</thead>
<tbody>
<tr>
<td>A1</td>
<td>550 ± 6</td>
<td>63</td>
<td>4.2 ± 0.2</td>
<td>35</td>
<td>15 ± 3.0</td>
<td>17 ± 3.4</td>
<td>21.4</td>
</tr>
<tr>
<td>A2</td>
<td>550 ± 6</td>
<td>67</td>
<td>4.2 ± 0.2</td>
<td>40</td>
<td>16 ± 3.2</td>
<td>28 ± 5.0</td>
<td>32</td>
</tr>
<tr>
<td>A3</td>
<td>550 ± 6</td>
<td>73</td>
<td>4.2 ± 0.2</td>
<td>35</td>
<td>14 ± 2.8</td>
<td>38 ± 6.6</td>
<td>21.4</td>
</tr>
<tr>
<td>B1</td>
<td>250 ± 7</td>
<td>130</td>
<td>4.3 ± 0.2</td>
<td>33</td>
<td>13 ± 1.4</td>
<td>23 ± 4.6</td>
<td>15.4</td>
</tr>
<tr>
<td>C1</td>
<td>250 ± 5</td>
<td>170</td>
<td>6.4 ± 0.2</td>
<td>31</td>
<td>6.7 ± 0.9</td>
<td>12.7 ± 1.5</td>
<td>7.3</td>
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</tbody>
</table>

*Calculated from eq. (3) (Vangbo et al).
Figure 1. Schematics of the center actuation experiment. In (a) beam geometry, including the approximated rectangular area section of the beam. In (b) water meniscus formed at the contact between the tip and the surface is sketched. For the experiments we assumed a tip radius $R_t$ of 8 nm, $R_2 = 5$ nm and $R_1 = 2.5$ nm. In (c) SEM images of beam C1 in up-buckled position and corresponding zoom out, showing the entire implanted area. In (d) SEM side view (90° tilted sample) of doubly clamped beam B1 in down buckled configuration. In (e) AFM topography and correspondent height profile of beam B1 up buckled.
Figure 2. (a) Force distance curve in contact mode during typical mid-span actuation experiment on beam A2, at high humidity (RH = 35%). The typical Z-shaped curve allows identifying different stages during actuation: (1) the tip comes into contact with the beam that is up-bend, (2) once the critical value of normal force ($F_{crit}$) is reached, snap through is triggered and the beam deforms to end in the down-bend stable configuration. The last branch of the curve is the elastic bending of the beam. During snap through the contact between tip and beam is never lost thanks to the presence of the water meniscus. From each curve is possible to identify certain parameters as indicated in (b). Apart from $F_{crit}$, $F_{min}$ is the lowest negative force detected, close to the value of the adhesion force. Note that the distance between the jump-to-contact and the beginning of elastic bending matches with twice the distance with respect to the horizontal plane ($2h_0$). In panel (c-e) actuation experiments in contact mode (only approach curves) for different beam geometry: type A (c), type B (d) and type C (e). RH in all experiments was set at 35%.
Figure 3. (a) Force-distance curves during mid span actuation on beam A2, tip retracts before completing the snap through, triggered at 60 and 150 nm displacement respectively. RH=35%; Z speed 1µm s⁻¹. The actuation can be reversed at any time, showing that the tip and the beam are bound during the entire cycle. (b) The first three buckling modes for doubly clamped beam. In panel (c-d) two solutions, Q= 2.5 and Q = 3, of the curved beam normalized force-displacement relation as calculated by Qiu and coworkers (ref. 10). F1 depends on Q while F2 and F3 are straight lines that do not depend on Q. For each solution we also reported the corresponding normalized experimental data, in this case for beam A1. In both cases experimental data overlap with F1 in the first part, and then follow a straight line with negative slope close to F3. Since F3 exists only with the second mode constrained, it is likely that the meniscus binding suppress the second mode. (e) Schematics of peak force scan at constant load and corresponding AFM height and deformation map of beam A1 in up-buckled position; brighter areas in deformation map correspond to areas with lower apparent stiffness, where performing actuation requires a minimum load (circled points in the sketch). See SI for further details.
Figure 4. (a) F-d curves in dynamic mode (only approach) during actuation at lower humidity. The curve acquired at 8% RH (blue color) presents a contact loss at around 125 nm displacement as confirmed by the amplitude signal acquired simultaneously. As F-d curves are acquired in dynamic mode, amplitude damping due to contact and meniscus formation is clearly visible in the amplitude channel. Contact loss is also present at higher humidity (RH = 20%); as expected it is placed at lower force values, the meniscus holds almost until the end of snap trough process. Interestingly, once the contact between tip and beam is lost the actuation proceeds towards its end as described in panel (b) where all stages of a switch between up and down buckling are described, separated by a dashed red line. Given the low humidity (RH < 8%) the retract motion leaves the beam unperturbed, as the meniscus is not able to pull up again the beam to its original position.


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