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An experimental study for characterization of size-dependence in microstructures via electrostatic pull-in instability technique

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ABSTRACT: This paper experimentally investigates the size-dependent effective Young's modulus $\left(E_{eff}\right)$ of Aluminum (Al) clamped-clamped microbeams using electrostatic pull-in instability technique. The study presents an experimental characterization of the so-called "length scale parameter" in couple stress theory and surface elasticity. The E_{eff} is retrieved from the measured pull-in voltage, of the clamped-clamped beams with different dimensions, via an electromechanically coupled equation. Measurement results show a strong size-dependence of E_{eff} for the Al beams in small sizes. The Young's modulus increases monotonously as the beams become thinner. The experimental observations are consistent with the published modelling results of the size effects, in which couple stress theory and surface elasticity theory are taken into consideration. The presented experimental method has substantial advantages such as precise adjustable magnitude of the non-contacting force and a lower cost over the other approaches used for characterization of micro/nanoelectromechanical systems. This simple and reproducible method can be extended for characterization of various materials with different sizes and boundary conditions.

The behavior of a wide variety of micro/nanoelectromechanical systems (M-NEMSs), such as actuators and sensors, with scales on the order of hundreds of nanometers depends on the stiffness of the components used. In these applications, it is important to have a fundamental understanding about the mechanical properties of the constituent materials, as these properties enormously affect frequency and deformation responses of the systems. The stiffness (EI) and Young's modulus (E) of a micro/nanostructure in bending and tension, respectively, are probably the most important mechanical properties. Continuum mechanics perception about the quantity E, which appears in both bending and tension, defines it as size independent. However, the effective stiffness of micro/nanostructures like wires, beams, and plates is different from those predicted by standard Hooke's law and Euler-Bernoulli theory. Experiments [1-7] revealed size dependency of mechanical behavior of structures when the size of the structure goes lower than

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the so-called critical size of the material. As evidenced by experimental investigations [1, 7-24] the effective Young's modulus depends not only on the thickness variation, but also on structural stiffness and residual stresses. These experimentally evidenced differences can indeed be explained by carefully accounting for surface effects and couple stresses [25]. Despite significant research in this area [1-7], still, there is not enough knowledge about the amount of the length scale parameter (LSP), surface elasticity and residual stress of different materials which are fabricated in different fabrication processes and/or subjected to different boundary conditions. An attempt was made by Sadeghian et al. [8] in using the pull-in technique to investigate the size effect, and it was concluded that the method is capable of studying the size effect, however they dealt with clamped free beams which do not experience the residual stresses. Besides, they did not consider an important factor that is the LSP appearing in couple stress theory. So, this paper is a step forward to shed more light on the individual, structurally isolated micro/nanostructures by providing experimentally measured data using electrostatic pull-in instability technique to determine the size-dependence of effective Young's modulus $\left(E_{eff}\right)$ for Aluminum (Al) clamped-clamped beams considering all the important factors, i.e. LSP in couple stress theory, surface elasticity and the residual stress effect.

While working on the submicron scale, loading methods are important to describe the elastic behavior of the structure. Due to the applications of bending mode in sensing [26-28] and also high sensitivity to surface elasticity [6, 29], research groups have focused on the characterization of micro/nanostructures especially E_{eff} in the bending mode [4]. In-plane bending via pull-in tests is used in this paper to experimentally investigate microbeams mechanical properties which are actuated by the electric field. In double clamped microbeams, constraints may impose a pre-loading caused by residual stress. Experimental characterizing of microbeams and extracting the LSP, surface elasticity and residual stress are goals of this paper. The order of magnitude for the obtained values for Al, as a metallic material, demonstrated a good agreement with previously measured experimental results for other types of metals such as Pb and Ag [25]. Of specific relevance to this work are the results of Wong et al. [30] who experimentally measured the elastic behavior of nanostructures. Poncharal et al. [19] measured the stiffness of carbon nanotubes, dynamically. These experiments showed that Young's modulus changes as the thickness, of the beams or the plates, or diameter of the wires or the tubes, reduces, revealing elastic properties size dependency at the submicron scale. A lot of theoretical work including mathematical modeling like atomistic modeling [15, 20] or the works related to continuum theory modifications [16, 17] can be found in the literature. There are also computer simulations such as empirical atomistic models of the covalent bond between carbon atoms which implemented to extract the uniaxial compressive and tensile response of carbon nanotubes [18, 31, 32]. Surface stresses can alter the elastic behavior of micro/nanostructures [33, 34] and modify the tensile response of micro/nanostructures. Robertson et al. [18], computed Young's modulus like quantity for carbon nanotubes which is dependent on the structure's diameter. They concluded that Young's modulus of the nanotubes reduces while they became smaller. On the other hand, as it is proved by other experiments [1, 17, 19, 21-24] E_{eff} can also show the opposite dependence with thickness, i.e. increase of stiffness due to size reduction or no dependence on the size effect [11, 30].

Atomic force microscopy (AFM) bending measurements and resonance frequency response [6] are the most popular approaches to extract E_{eff} which suffer from mass-loading effect, with difficulties in implementation and high cost of the facilities. In the latter case, i.e. resonance frequency response, E_{eff} can be obtained from fitting frequency response to the vibration equation of the structure [6]. The frequency response not only depends on the stiffness but also the mass of the structure. So, dissociation of the mass effect from the stiffness effect is challenging because of surface contamination, additional surface layers like oxide layer, etc. [35]. This can potentially increase dissipation and degrade sensitivity of the resonance frequency response method. AFM test provides high displacement resolution via applying direct contact force on the structure. The most popular AFM method is done by single-point measurements. Reasons like tip slippage [13] and torsional problems of the probe [11] can often lead to uncertainties in interpretation of the experimental results. Using multipoint measurements in AFM can alleviate these inaccuracies somehow but still the remained uncertainties are considerable [36].

Neglecting the fringing effect, bending in the pull-in method is due to an electrostatic force. The electrostatic force is a body force and therefore it is uniform and consistent between the electrodes. Unlike the resonance frequency method, the pull-in method is a mass-independent approach (see Eq.1) that is simply a function of the effective stiffness of the beam and the capacitive force. All these points make the pull-in method a unique and precise method, despite having its limitations. Pull-in method is constrained by the material type itself which should be a conductive material. This limitation however can be counted for AFM method somehow, as stiffness limitation of the probes in this method will not allow using this method for structures which are stiffer or softer than the probe itself.

Furthermore, from the fabrication point of view, due to the stiction phenomena, the release of the capacitive structures with small gaps (lower than 1 micron) can be potentially challenging. This problem arises even more when trying to produce clamped-free devices like cantilevers due to unavoidable strong liquid capillary force and stress-induced effects in the structure before doing the release process. In these cases, it is very likely, the free end of a cantilever adhere to the fixed electrode or even the substrate, permanently. Using supercritical drying or layer passivation with a lubricant, this problem can be somehow tolerated but not in the low-stiffness structures. In contrast, for a production of low mass capacitive mechanical structures, doubly clamped structures offer a wide range of flexibility and can deal with the problem quite well. So, we have focused on clamped-clamped beams in this work. In these structures, the stuck stress is called residual stress.

Experimental error is in the nature of scientific measurements and it is very important to minimize the error sources as much as possible. Some errors are due to inherent limitations in the equipment or techniques. It is persuasive that the pull in method is promising in reducing the source of errors compared to the other methods mentioned above. In addition, various read-out systems such as a simple electrical circuit for detecting system voltage (see Fig. 1), can be easily used to get the sharp response of pull-in instability due to a short circuit. Pull-in experiment is used by research groups to extract mechanical behavior of the structures [37] and the obtained results are quite promising but the method has never been used for extracting LSP and surface elasticity which we did it here in this study. In this method, a voltage is applied between two electrodes, one electrode is fixed and the other is not. The movable electrode is pulled down due to the electrical force. The static pull-in voltage is determined by coupling the mechanical behavior of the structure to an electrostatic pressure term and finding the lowest voltage at which the system is unstable and the movable electrode snaps to the fixed one. Through the general differential equation of forced vibration of a structure in static mode considering electromechanical interaction, the effective stiffness ($E_{eff}I$) can be stated as follow:

$$E_{eff}I\frac{\partial^4 w}{\partial x^4} = \frac{\varepsilon_0 V^2 A}{2g_{eff}^2} \tag{1}$$

This equation can be used to calculate pull-in instability accurately, where I is the cross-sectional moment of inertia, ε_0 is the permittivity of vacuum, and V is the applied voltage. E_{eff} is presented by Abazari et al. [25] and is equal to $E_{bulk} - 6C_s\left(\frac{1}{h}\right) + 24\frac{E_{bulk}}{1+\nu}\ell^2\left(\frac{1}{h}\right)^2 + \frac{3}{10}\sigma_0\left(\frac{L}{h}\right)^2$, where $E_{bulk}, \nu, C_s, \ell, \sigma_0$, and h are standing for Young's Modulus for the bulk material, Poisson's ratio, surface elasticity, LSP, residual stress and thickness of the beams. This model is not a perfect physical law but it is an approximation which is broadly accurate, see the results presented in Table 2 of [25], where it is shown the discrepancies between experiments and the model predictions nearly vanish while different materials behavior is studied with this model. The Young's Modulus as a material property can be expressed as a sum of terms resulting from independent contributions ($E_{eff} = E_{\text{bulk}} + E_s + E_c + E_{other}$). Here, E_{bulk} is the general Young's Modulus of the bulk material, E_s is an independent contribution caused by the surface, and E_c is the contribution caused by microstructure of the material. E_{other} represents the other contributions which can be boundary conditions that we have considered here as residual stress and dislocations and/or other crystallographic defects. g_{eff} in Eq.1 is the initial effective gap between the movable electrode and the fixed one. Clamped structures would likely buckle due to compressive stress which would change the nominal gap (g_n) of the mechanism. So, if we replace the stress effect with eccentric loading in structures and let the beams to buckle both in-plane (this can make the gap bigger or smaller) and out of the plane, the effective gap can be found using Equation (2):

$$g_{eff} = \sqrt{{g_n}^2 \pm g_n \frac{3L^2|\sigma_0|}{2hE_{bulk}} + \left(\frac{3L^2\sigma_0}{4bE_{bulk}}\right)^2} \tag{2}$$

Due to the nonlinearity of Eq. 1, solving it is complicated and time-consuming. The direct application of either the Galerkin method or the finite difference method will produce a set of nonlinear algebraic equations. To avoid this problem, the step-by-step linearization method (SSLM) [38] is applied followed by the Galerkin method to linearize and solve the obtained linear set of algebraic equations. By solving Eq.1, the instability point, i.e. pull-in voltage, can be extracted. The pull-in voltage was experimentally measured for various beams with different thicknesses and lengths but the same aspect ratio (AR=$\frac{L}{h}$=50). The E_{eff} data versus 1/h of the beams were fitted to Eq.1, which then was solved by the SSLM. From the solution, the values for each parameter were determined. In calculations, E_{bulk} assumed to be 70 GPa for Al material. The Al beams, with $2\mu m$ thickness that was deposited by sputtering on the Si wafer, were used for this investigation were made by standard fabrication processes using silicon on insulator (SOI) wafers. As shown in Fig. 1(a & b), the measurement setup consists of a source meter (KEITHLEY 2400) and a manual testing probe station (KARL SUSS PM5). Figure 1(b) also shows the scanning electron microscopy (SEM) of the fabricated beams. The pull-in point was measured using step by step increasing of the voltage difference between the movable beam and the fixed part (1 Volt per step). A LabView interface is used to determine the pull-in voltage (voltage (V) & current (I) plot).

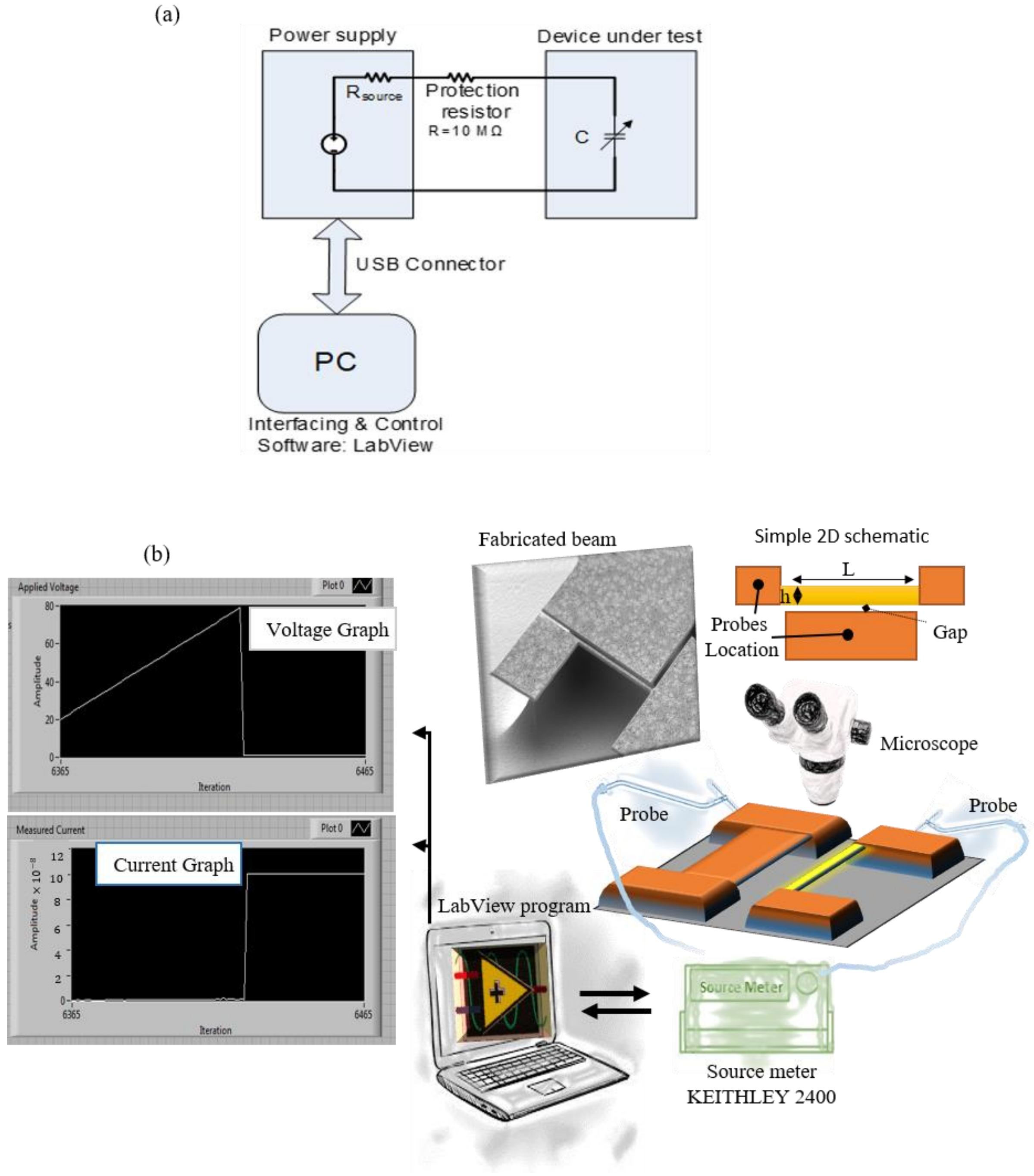
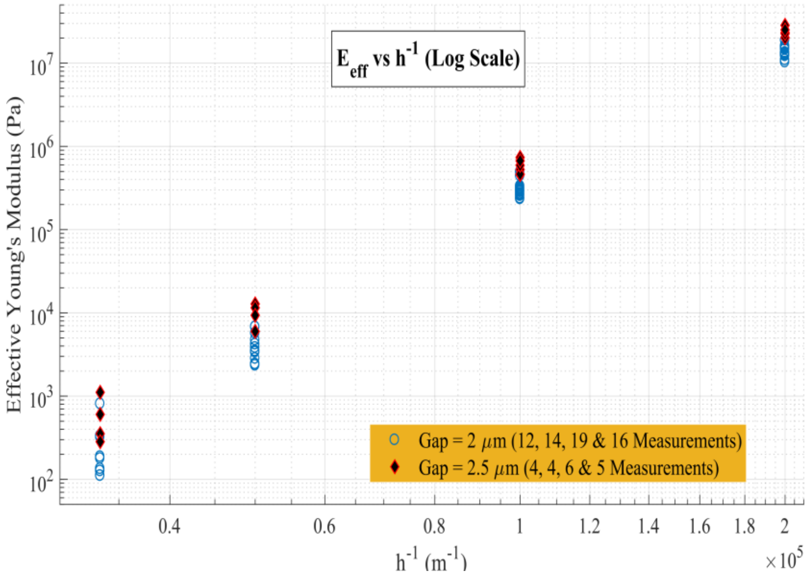


FIG. 1. Schematic view of the measurement setup

Figure 2 (a) shows the extracted E_{eff} as a function of 1/h (where log scale is used to better represent the data) and Fig. 2 (b) shows the extracted parameters for the measurements. Two different sets of experiments with different gaps (2μm and 2.5μm) were carried out to check the repeatability and reliability of the experimental results. Table I provides the geometrical dimensions and the pull-in voltage measurements. Comparing the results for the different gap sizes shows that the performed measurements are both repeatable and reliable for the samples with different thicknesses, lengths, and shapes.



Gap 2.5	
LSP	33 nm
Cs	29.5 N/m
Residual stress	-93.3MPa

Gap 2	
LSP	25 nm
Cs	16.3 N/m
Residual stress	-93.3MPa

(a) (b)

FIG. 2. (a) The size dependence of the E_{eff}. The markers (the red diamond and the blue circle markers are for structures with 2.5 and 2 micrometer gap, respectively) represent the E_{eff} for tested clamped-clamped beams (b) the parameters extracted from fitted results

TABLE I. Geometrical dimensions and pull-in voltage average measurements and their associated standard of deviation (The last column is showing the percentage error which is calculated by division of Average values by Standard Deviation values)

Gap Size (μm)	Nominal dimensions (μm) Width= 2		Pull-in voltage measurements		
	h	L	Average pull-in voltage (V)	Standard Deviation	Error (%)
2	5	250	129.2	± 3.1	2.4
	10	500	93.0	± 4.0	4.3
	20	1000	64.7	± 4.5	7.0
	30	1500	52.9	± 5.2	9.9
2.5	5	250	182.0	± 2.9	1.6
	10	500	131.7	± 3.9	2.9
	20	1000	94.3	± 4.6	4.9
	30	1500	77.8	± 5.0	6.4

Serious challenges can be raised while investigating size dependency either theoretically or experimentally and yet this is the bottleneck in the way of research trying to predict the performance of micro/nanosystems. There are intrinsic and extrinsic defects which are induced because of physical laws (like Gibbs free energy law) and processing condition and/or environmental effects, respectively. These defects cause structural imperfections which can dramatically change the material properties. As a result, the scatter in E_{eff} observed during the experiments is due to dislocations, linear or planar defects, crystallographic defects [4, 20, 39] and also different grain morphology and impurities that exist both in the surface and the bulk material.

In summary, pull-in instability measurements provide a reliable and accurate experimental measurement of E_{eff}. Experimentally extracted LSP, surface elasticity and residual stress for the fabricated Al clamped-clamped beams were about 30 nm, 23 N/m and -93.3 MPa (minus stands for compressive stress), which are representing crystallographic, surface and preloading effects of micro/nanostructures, respectively. This compendium experimental letter is useful to study the size effect in micro/nanostructures with a wide variety of materials. The presented technique offers a simple solution for the challenging and long-needed LSP characterization in the couple stress theory. This study suggests that the order of the LSP value is in nanometer scale for Al, and a similar value range is expected for other metallic materials.

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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