High Confidence Level Calibration for AFM Based Fracture Testing of Nanobeams

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Abstract

When designing micro- or nanoelectromechanical systems, (MEMS and NEMS), it is important to consider whether structural elements will withstand loads experienced during operation. Fracture behavior at length scales present in MEMS and NEMS is much different than at macro- and mesoscopic scales. Due to a smaller probability of crystal defects and a high surface to volume ratio, fracture is controlled by surface characteristics rather than volumetric ones. Prior measurements using doubly clamped Si beams loaded with an atomic force microscope (AFM) showed that fracture of Si nanobeams is highly affected by surface roughness [1] and oxidation [2]. In experiments of this type, calibration of the system, particularly the AFM cantilever stiffness, is critical to the accuracy of both the force and displacement results. A new set of experiments are underway in which the tests are performed by adapting a direct, traceable method for calibrating the AFM cantilever stiffness [3]. The improved calibration should not only improve the accuracy of the strength results but will allow linear stiffness measurements of the sample to be used to back out sample thickness, a key parameter in interpretation of the data.

Introduction

The rapid development of micro- and nanoelectromechanical systems (MEMS and NEMS) necessitates the ability to predict and control strength of micro- and nanoscale structures. Previous results have shown that these types of structures can have strengths approaching the ideal strength of the material and that strengths are strongly dependent on both the surface roughness [1] and surface oxidation [2]. These tests were performed using doubly clamped silicon beam structures which were under-etched so that they were freely suspended across a trench. An atomic force microscope (AFM) cantilever was then used to deform the beam structures until failure. Because the AFM cantilever deforms as well as the beam, calculating the stress at failure relies explicitly on knowing the stiffness of the AFM cantilever.

It is often necessary to know the stiffness of an AFM cantilever when one is interested in quantifying the forces between the tip and the sample. Several methods have been suggested for measuring stiffness. If the length L, width w, elastic modulus E, density ρ, and resonant frequency f_0 are known to high enough precision, the stiffness may be calculated using standard beam theory by [4]

$$k = 2\pi^3 w \left(f_0 L \sqrt{\rho}\right)^3 / \sqrt{E} \,. \tag{1}$$

The Sader method involves calculating stiffness by observing the effects of dynamic loading and damping by the viscous fluid surrounding the cantilever (typically air) on the frequency and quality factor of resonance [5]. Cleveland *et al.* developed a method in which a small mass is added to the end of the cantilever and the stiffness is calculated from the change in resonant frequency [4]. Hutter and Bechhoefer suggested relating the stiffness to magnitude of thermal vibrations using the equipartition theorem by [6]

$$1/2k \left\langle z_t^2 \right\rangle = 1/2k_B T \,, \tag{2}$$

where $\left\langle z_t^2 \right\rangle$ is the mean square displacement of thermal motion, k_B is the Boltzmann constant, and T is the absolute temperature. Butt and Jaschke added a correction factor to Equation (2) by considering the shape of each mode of vibration [7]

$$k \left\langle z_t^2 \right\rangle = 0.817 k_B T \,. \tag{3}$$

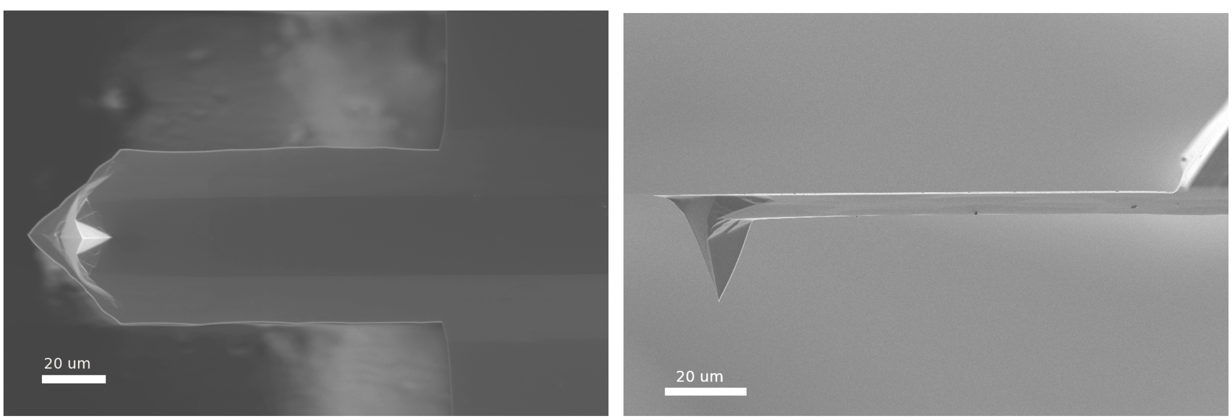
This method is used for automated calibration of cantilever stiffness in many commercial AFM systems [8]. Ohler has provided a concise review of these methods and a direct comparison of the results of each for a range of cantilever stiffnesses [9]

Although these techniques are sufficient for most AFM applications, they are poorly suited to the strength measurements developed by Alan *et al.* [1, 2]. For typical AFM applications, a fairly compliant cantilever is used so that weak sample-tip interactions can be observed. In the case of strength measurements, the cantilever must be much stiffer. The cantilevers used in the beam fracture experiments have stiffnesses of around 200 N/m - 250 N/m. In contrast, the Sader, added mass, or thermal calibration techniques are typically used to calibrate cantilevers with stiffness of 0.01 N/m - 35 N/m. To have confidence in the resulting fracture strength data, the stiffness of the cantilever should be measured with a method well suited to very stiff cantilevers.

In this paper, we will compare two methods, that used by Alan *et al.* [1, 2] and a new one based on work by Gates *et al.* [3, 10]. In the method of Alan *et al.* , the length, width, and tip position of the cantilever was first quantified using a scanning electron microscope (SEM). The cantilever thickness was then estimated by comparing the measured resonant frequency of the cantilever to that found using a variable thickness thickness finite element model using density and elastic modulus values from the literature. The method based on the work by Gates *et al.* involves measuring the stiffness of a reference cantilever using an instrumented nanoindentor. That reference cantilever can then be used to measure the stiffness of the cantilever for use in beam fracture experiments. Following a more thorough description of each of these methods, we will compare stiffness values obtained with each method and the accuracy of each.

Methods

The resonant frequency method used by Alan *et al.* starts with taking an SEM image of the cantilever to be calibrated with the cantilever oriented so that thickness direction is normal to the plane of the image as shown in Figure 1a. Using this image, the length and width of the cantilever can be obtained as well as the position of the tip. These dimensions are then used to create a finite element model such as that shown in Figure 2. Because the thickness of the cantilever could not be obtained directly from the SEM image, the thickness of the finite element model is varied until the resonant frequency of the model matches the experimental resonant frequency. At this point a static load can be applied to the tip of the cantilever in the finite element model. By dividing that load by the resulting deflection one obtains the cantilever stiffness.



(a) (b)

Figure 1: (a) SEM image of AFM cantilever from which cantilever dimensions would be taken to construct a finite element model from which one can obtain the stiffness. This is referred to as the test cantilever in the text. (b) SEM image of the same AFM cantilever from the side. The tip height and cantilever may be obtained from this image.

The second method is drawn from the methods of Gates *et al.* who have developed a method in which the cantilever to be calibrated displaces a reference cantilever of known stiffness [10]. In order to accommodate a range of cantilever stiffnesses, Gates *et al.* suggests using an array of reference cantilevers which span a range of stiffnesses. Unfortunately we again run into a problem that the cantilever to be used for beam fracture experiments is far stiffer than any of those used by Gates *et al.* . Ying *et al.* have also developed a second method in which an instrumented

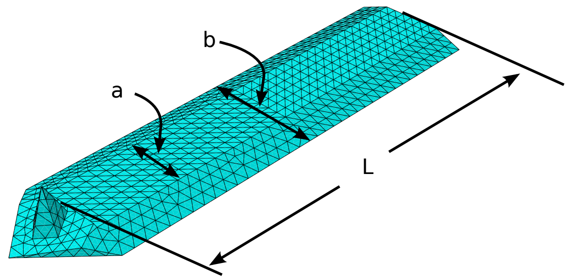


Figure 2: A finite element model of an AFM cantilever with the dimensions obtained from an SEM image such as that shown in Figure 1a.

nanoindentor is used to directly measure cantilever stiffness [3]. This has the disadvantage that each cantilever that would be used for nanobeam fracture tests must be calibrated with the nanoindentor and the nanoindentor is not at the same facility where the rest of the tests are to be done. The method we have developed combines the two methods proposed by Gates *et al.* .

The new stiffness calibration method uses a batch of reference cantilevers which are only slightly more compliant than the cantilevers to be used for fracture testing. An SEM image of one of these reference cantilevers is shown in Figure 3. The stiffness of these reference cantilevers is measured directly with an instrumented nanoindentor. Each of these reference cantilevers may then be used to calibrate the stiffness of a large number of other cantilevers. Notice that the reference cantilever shown in Figure 3 does not have a tip. Since the reference cantilever is used only to calibrate the stiffness of other cantilevers, it will never interact with a sample surface and so a tip is not needed. A tip would only get in the way when used with the instrumented nanoindentor and with other other cantilevers.

To use the reference cantilever to calibrate the stiffness of a second cantilever, the reference cantilever is placed on the stage of an AFM and the cantilever to be calibrated is mounted normally. The test cantilever is first pushed against the bulk silicon at the base of the reference cantilever. This bulk silicon is considered to be approximately rigid and so the test cantilever deflects an amount equal to the displacement of the AFM head. The output of this is data in volts and nanometers as shown in Figure 4. The horizontal axis, in nanometers, is the displacement of the base of the cantilever as applied by the AFM head. The vertical axis, in volts, is proportional to the deflection of the tip of the test cantilever relative to its base. The trend starts out flat as the cantilever approaches the surface. Once the tip contacts the surface, the deflection signal begins to increase. From this we extract a value called the sensitivity S_1 which is the inverse of the slope of this plot after the tip has contacted the surface. It has units of nm/volt and relates the signal in volts to the deflection of the cantilever tip relative to the cantilever base.

The test cantilever is next aligned over the location at which the reference cantilever was calibrated and a second force curve is performed. The test cantilever pushes on the reference cantilever and both deflect, as shown in Figure 5. Because both cantilevers are deflecting, they act as springs in series and so the AFM head must travel through a greater distance to produce the same tip deflection relative to the cantilever base. This means that the slope of the resulting plot is less. Although it is not a true sensitivity, we will call the inverse of this slope S_2.

In order to find the cantilever stiffness k in terms of S_1, S_2, and the reference cantilever stiffness k_{ref}, note that

$$\delta = S_1/V \,, \tag{4}$$

where δ is the deflection of the test cantilever and V is the deflection signal in volts. Similarly,

$$S_2 = \Delta/V = (\delta_{\text{ref}} + \delta)/V \,, \tag{5}$$

where δ_{ref} is the deflection of the reference cantilever and Δ is the head displacement when the test cantilever is in contact with the reference cantilever and is the sum of the displacements of both cantilevers. Eliminating the deflection signal V from Equations (4) and (5),

$$S_2 = \left(\frac{\delta_{\text{ref}} + \delta}{\delta}\right) S_1 \;\rightarrow\; \frac{\delta_{\text{ref}}}{\delta} = \frac{S_2 - S_1}{S_1} \,, \tag{6}$$

Since the two cantilevers are in static equilibrium, we also know that

$$k_{\text{ref}}\delta_{\text{ref}} = k\delta \;\rightarrow\; k = \frac{\delta_{\text{ref}}}{\delta} k_{\text{ref}} \,. \tag{7}$$

Finally, in order to get the cantilever stiffness k in terms of S_1, S_2, and k_{ref}, we substitute Equation (7) into Equation (6) to get

$$k = \left(\frac{S_2 - S_1}{S_1}\right) k_{\text{ref}} \,. \tag{8}$$

The same cantilever was calibrated with both the reference cantilever method and the resonant frequency/finite element model method. The calibrated cantilever was an uncoated TAP525 from Bruker AFM Probes, model number MPP-13100-10 with a nominal stiffness of 200 N/m. The reference cantilever was an uncoated AppNano ACL-TL cantilever with a nominal stiffness of 45 N/m, although the group of reference cantilevers was handpicked to have a higher than average stiffness of at least 60 N/m. The nanoindentation was done with a Hysitron Triboindentor at the National Institute for Standards and Technology[1] and the AFM work was done with a Dimension Icon 3100. All

[1]Certain instruments and materials are identified to adequately specify the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or

finite element analysis was done using ABAQUS. For the finite element simulations, the density of silicon was set to 2.329 g/cm^3 and the C_{11}, C_{12}, and C_{44} elastic constants were set to 166 GPa, 64 GPa, and 79.6 GPa respectively. The crystal orientation was set so that the top and bottom surfaces of the cantilever were (100) planes and the long axis of the beam was in the [110] direction. This orientation was confirmed in the physical cantilevers using Laue back-reflection of X-rays.

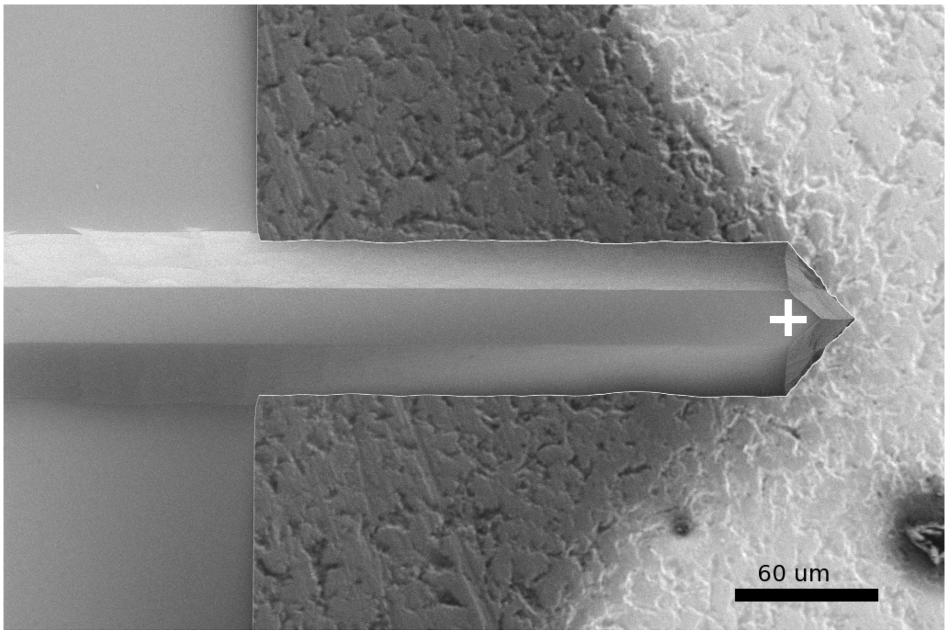


Figure 3: SEM image of a reference cantilever used to calibrate the stiffness of cantilevers used for nanobeam fracture tests. The white cross represents the location at which the cantilever is calibrated.

Results

The stiffness of the reference cantilever was measured with the instrumented nanoindentor ten times, producing an average value of 79.3 N/m with a standard deviation of 0.467 N/m. Using these values in the reference cantilever method, the stiffness was found to be 233 N/m. Using the finite element model with the model thickness adjusted so that the resonant frequency of the model matched the measured resonant frequency, the stiffness was calculated to be 183 N/m. A second stiffness calculation using the finite element model was also performed except the thickness was measured directly from a side view SEM image, Figure 1b. In this case, the resulting stiffness was 231 N/m.

The Hysitron nanonindentor calibration has an uncertainty of about 3% and repeated measurements with the nanoindentor probe repositioned each time showed an uncertainty in repeatability of about 2%. These combine to give an uncertainty in reference cantilever stiffness of 3.6%. The uncertainty in placement of the test cantilever is about

instruments identified are necessarily the best available for the purpose.

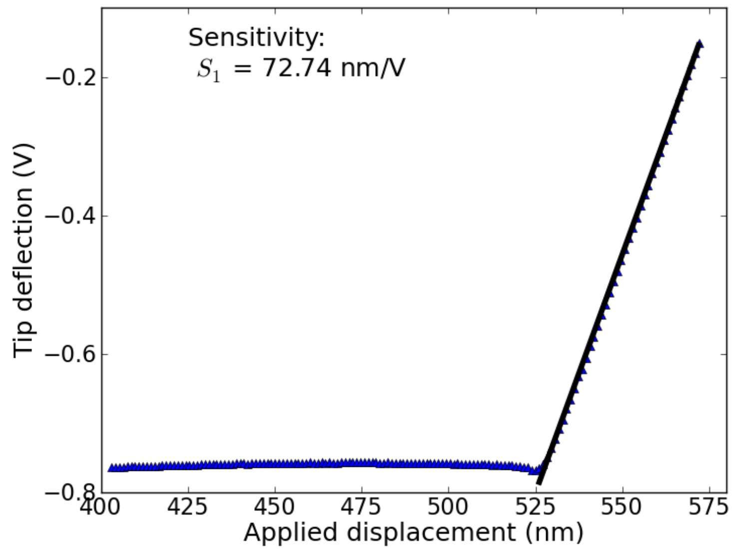


Figure 4: Plot showing the measurement of the AFM cantilever sensitivity S_1 as the cantilever is pushed against a hard surface.

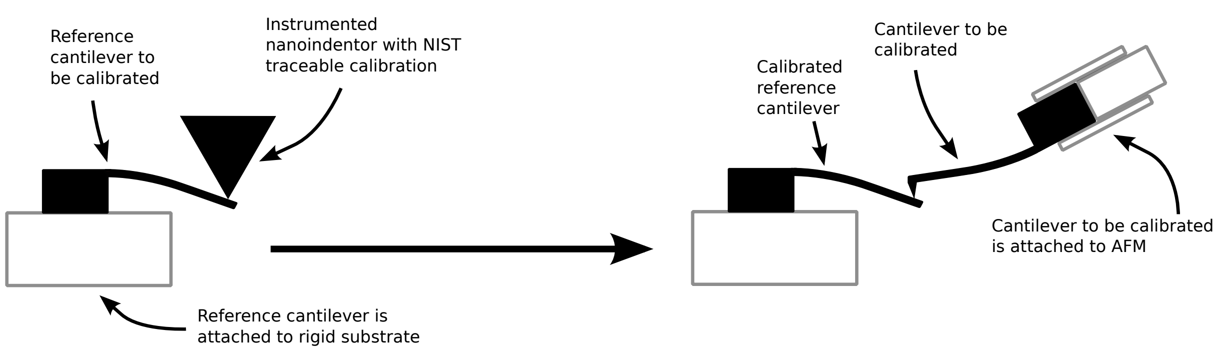


Figure 5: The reference cantilever is first calibrated using a NIST traceable instrumented nanoindentor. During calibration of the test cantilever, it is pushed against the end of the now calibrated reference cantilever. Because the stiffness of the reference cantilever is known, the stiffness of the test cantilever can be calculated.

2%. Combining this with the uncertainty in reference cantilever stiffness gives a potential error for the reference cantilever method of 4.1%.

In the resonant frequency method, errors in measurements of cantilever dimensions lead to errors in resonant frequency which, which produces an error in thickness, which ultimately results in an error in stiffness. Notice from Figure 1 that the cantilever may be partitioned into three parts. The first and largest part, which we will call the arm of the cantilever, is from the base to the tip and has a constant trapezoidal cross section.The pyramidal tip which interacts with the sample is a second part and the third is the somewhat irregularly shaped triangular point at the end of the cantilever. Because the mass of the second and third parts are difficult to determine from Figure 1a and they do not contribute much to the cantilever stiffness, we will lump them together into a single mass m In a similar fashion as the added mass method [4], the resonant frequency may be expressed as

$$\omega = \sqrt{\frac{k}{m + M^*}}\,, \tag{9}$$

where k is the stiffness of the finite element model before the thickness is adjusted, m is the combined mass of the cantilever tip and the triangular point region, and $M^* = 0.24M$ where M is the mass of the cantilever arm. M and k can be expressed as

$$M = \rho L t \left(\frac{a+b}{2}\right)\,,\; k = \frac{Et^2(3ab)}{12l^3}\,, \tag{10}$$

where ρ is the density of silicon, t is the thickness, L is the cantilever length, and a and b are the short and long parallel sides respectively of the cantilever's trapezoidal cross section. These dimensions are illustrated in Figure 2. From Equation (9), uncertainty in the resonant frequency ω as calculated using the finite element model relates to uncertainties in m, L, a, and b by

$$\begin{aligned}\left(\frac{\delta\omega}{\omega}\right)^2 = \frac{1}{4}\left(\frac{m/M^*}{1+m/M^*}\right)^2\left(\frac{\delta m}{m}\right)^2 + \left[\frac{9}{4} + \frac{1}{4}\left(\frac{M^*/m}{1+M^*/m}\right)^2\right]\left(\frac{\delta L}{L}\right)^2 + \\ \left[\frac{1}{4}\left(\frac{1}{1+b/3a}\right)^2 + \frac{1}{4}\left(\frac{1}{1+b/a}\right)^2\left(\frac{M^*/m}{1+M^*/m}^2\right)\right]\left(\frac{\delta a}{a}\right)^2 + \\ \left[\frac{1}{4}\left(\frac{1}{1+3a/b}\right)^2 + \frac{1}{4}\left(\frac{1}{1+a/b}\right)^2\left(\frac{M^*/m}{1+M^*/m}\right)^2\right]\left(\frac{\delta b}{b}\right)^2\,.\end{aligned} \tag{11}$$

If we make the approximation that m is small compared to M^*, then thickness is linearly proportional to resonant frequency and their uncertainties are equal. From Equation (10), we see that

$$\frac{\delta k}{k} = 3\frac{\delta t}{t} = 3\frac{\delta\omega}{\omega}\,, \tag{12}$$

where $\delta\omega/\omega$ can be found using Equation (11). Errors in length measurements using SEM are estimated to be at most 5%. Using measurements taken from SEM images such as those in Figure 1, $\delta m/m$ is estimated to be about 10%. Using these values in Equations (11) and (12), the maximum error in cantilever stiffness using the resonant frequency method is estimated to be $\delta k/k = 25\%$.

Discussion

It is immediately obvious that the stiffness calculated using the finite element with the model thickness adjusted by resonant frequency does not match very closely with that found using the reference cantilever. The fact that the finite element generated stiffness agrees with the reference cantilever method when the model thickness is measured directly by a side view SEM image suggests that the reference cantilever method is accurate. It is also apparent that if the thickness can be accurately determined the finite element model can produce good results as well. The arm region of the cantilever,extending from the base to the tip, has a very simple geometry which is easy to accurately represent in the finite element model. The pyramidal tip has a simple geometry but extends in a direction normal to the plane of the image and it is impossible to determine the height of this tip from Figure 1a. The thickness of various portions of the triangular point region at the end of the cantilever also cannot be determined directly from Figure 1a. The data indicates that the potentially large error in the mass of the triangular point and tip regions of the cantilever cause an error in resonant frequency. This leads to the model thickness being adjusted to the wrong value, which results in an incorrect cantilever stiffness. Additional analysis few the relative uncertainties and ac curacies of the two methods is in progress.

Summary

We have compared two methods for calibrating the stiffness of very stiff AFM cantilevers. The first method involved measuring cantilever dimensions in a SEM, finding the cantilever thickness by matching model resonant frequency to actual resonant frequency, and using the model to calculate stiffness. The second method involved measuring the stiffness of a reference cantilever using an instrumented nanoindentor and then using that as a transfer artifact to calibrate the stiffness of the test cantilever via the reference cantilever method. We have found that due to difficulties in accurately finding the volume of the cantilever tip, the method of finding thickness with resonant frequency does not result in a stiffness that matches with that found using the reference cantilever. By using a finite element model with a thickness obtained using a side view SEM image the model produces a stiffness very close to that of the reference cantilever method. Uncertainty for the reference cantilever method has been shown to be considerably smaller than that of the resonant frequency method.

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