STM/SEM METHOD FOR TESTING BENDING STRENGTH OF MEMS BEAMS

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ABSTRACT

The observed fracture strength of materials when used at NEMS and MEMS scales are generally higher than the bulk properties, particularly as the size decreases to nanometers. Such high strength offers the possibility of designing devices that sustain very high stresses, for example tensioned oscillators, or energy storage components. To study the fracture of micron scale cantilevers, we propose a new test method that makes use of an ultra high vacuum STM/SEM combination and digital image processing. The experiment consists of using a slightly blunted STM tip to load a cantilever beam. While deflecting the cantilever, a sequence of digital images of the deformed beam is acquired with the SEM, from the first load up to the point of fracture. The elastic modulus of the beam is determined before the fracture test in a separate experiment by use of resonant frequency measurement. The sequence of SEM images is processed to determine the fracture strength by first creating a (mathematical) model cantilever of the same size and cross section as the tested cantilever. Using large deflection beam theory, the deflected shapes of this imaginary cantilever are obtained for increasing loads. Synthetic images of the deformed model cantilever are formed by mapping onto the computer screen using projection angles and magnifications determined from a separate calibration test. The final step is to compare the synthetic images to the digital ones captured by the SEM. The applied load on the model beams is iterated to obtain the best fit with the processed SEM images. Once the load that causes the beam to fracture is known, the fracture strength can be easily determined. To demonstrate the procedures we present initial results for untreated silicon cantilevers as well as for samples heat treated in vacuum by applying resistance heating prior to testing.

1 INTRODUCTION

To properly design highly stressed components of micro- and nano-electromechanical systems (MEMS & NEMS), a designer must know the mechanisms and origins of fracture and must also know the fracture strength statistics of the materials under consideration. Silicon, in single or poly crystalline form or as a compound with other materials, is by far the most commonly used material in NEMS technology. Experiments and ab-initio [1] calculations show that the bulk elastic properties apply down to a length scale of several nm's. Thus, at least down to thickness of several nm, one need not be concerned with the elastic properties of single crystal Si. However, the elastic properties of polycrystalline and compound materials are dependent on the processing conditions, which in turn may depend on the size scale of the system being fabricated. Thus test methods for elastic properties are needed as well. In this work, however, we concentrate on measurements of fracture strength.

Single crystal silicon is brittle at room temperature, thus it is expected to fracture with no appreciable yielding. However, there is little agreement on what the failure strength of silicon is. The only commonly accepted point is that the failure strength changes with the size and shape of the sample. Hence we cannot apply bulk strength properties for silicon. For example, Namazu [2] found an increase in mean bending strength of cantilevers as the size scale reduces to nanometers. Considering that fracture is related to the presence of surface and bulk defects, higher strength values are expected for materials with lower defect density. Reducing the size scale is one way of reducing the probability that a component will contain a defect that will greatly reduce strength. An effective way to control the strength of silicon may be to modify its surface so as to diminish the density of surface defects. By means of surface treatment, and by modifying the chemical procedures in the fabrication process, it is possible to control the surface of the structures, and hence their failure strength.

In this paper we introduce a new method utilizing an ultra high vacuum STM/SEM combination and digital image processing to test the bending strength of micron size scale cantilevers. Advantages of this approach are that it allows the rapid testing of samples made from relatively simple procedures, it can scale down to beam lengths of approximately 10 microns and it allows us to visually observe the failure of the beam. Another important advantage of using the ultra high vacuum STM is that we can use it to heat treat and image our samples prior to testing. A disadvantage is that the load applied to the sample is not directly measured.

First we give an overview of the method, then we describe the calibration of the SEM and digital image processing employed. To demonstrate the method we describe initial results obtained from tests of commercially available AFM cantilevers before and after heat treatment.

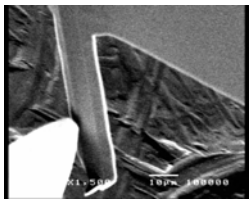
2 EXPERIMENTAL PROCEDURES

To perform our experiments we use the combination of an ultra high vacuum Scanning Tunneling Microscope and a Scanning Electron Microscope (JEOL UHV-4500). We use the STM both to load the samples as well as to image their surfaces prior to testing. The SEM is used to image the beams as they are deformed during testing.

In our experiments, after determining the elastic modulus by resonance frequency measurement, we employ a blunted STM tip to push against a cantilever beam using the coarse motion control of the STM, deflecting the beam up to the point of fracture, see Figure 1. Meanwhile, a sequence of digital images of the deflected cantilever is obtained with the SEM, the electron gun of which is pointing at the image observation chamber of the STM. Knowing the elastic modulus and geometrical properties of the beam, we can process the captured digital images using large deflection beam theory and obtain its fracture strength as explained below. Yet, before any image processing we discuss the calibration of the SEM and fabrication of blunted tungsten tips.

2.1 Calibration of the SEM

As mentioned earlier, the test is displacement controlled. During coarse stage control of the



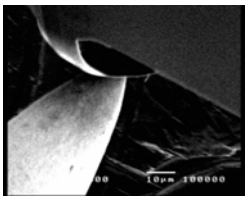


Figure 1: Deflection of a cantilever beam under loading by a blunted STM tip.

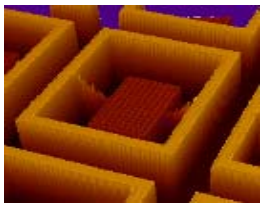
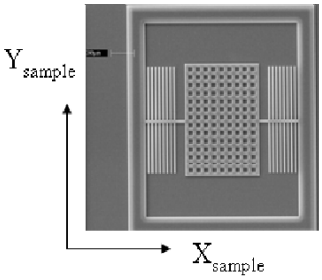
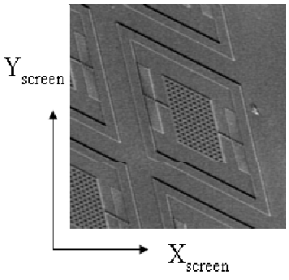


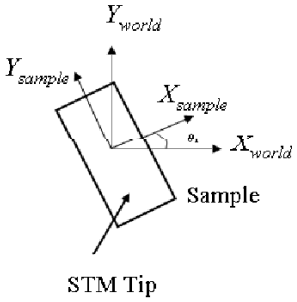
Figure 2: The calibration sample



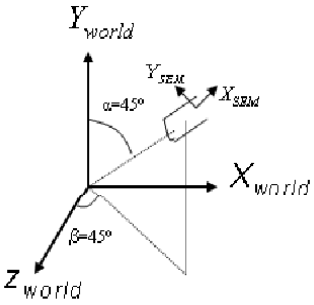
(a)



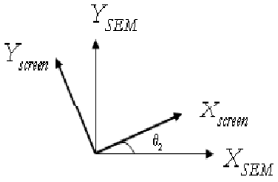
(b)



(c)



(d)



(e)

Figure 3: a) calibration sample in sample coordinates, b) calibration sample in screen coordinates, c) sample rotated around the STM tip, d) position of the SEM, e) screen vs. SEM coordinates.

STM used for the experiments, the deflected shape of the beam can be accurately determined by comparing SEM images which are 2D projections of the out-of-plane beam deflection onto the screen coordinates. To determine the actual beam deflection, the coordinate system in which the geometry of the sample and the deflections are defined must be related to the screen coordinate system. For this purpose we use the 3-D grid structure shown in Figure 2 for calibration.

The SEM electron gun is directed towards the sample at a 45 degree azimuth rotation and 45 degree vertical elevation. Once loaded in the high vacuum chamber, the sample faces the STM tip

perpendicularly. However, because of its position relative to the sample holder, it is generally subject to an unknown, in-plane rotation θ_1 around the tip. Moreover, we assume that the vertical and horizontal magnifications of the SEM may be different and that the screen axis is rotated around the centerline of the electron gun, by θ_2 (Figure 3).

Therefore, both coordinate systems can be related to each other by applying the following five transformation matrices:

i) $$R_{z1} = \begin{bmatrix} \cos\theta_1 & \sin\theta_1 & 0 \\ -\sin\theta_1 & \cos\theta_1 & 0 \\ 0 & 0 & 1 \end{bmatrix}$$

rotation of the sample around the STM tip

ii) $$R_y = \begin{bmatrix} \cos\beta & 0 & -\sin\beta \\ 0 & 1 & 0 \\ \sin\beta & 0 & \cos\beta \end{bmatrix}$$

rotation of the SEM in the horizontal plane, $\beta=\pi/4$

iii) $$R_x = \begin{bmatrix} 1 & 0 & 0 \\ 0 & \cos\alpha & -\sin\alpha \\ 0 & \sin\alpha & \cos\alpha \end{bmatrix}$$

vertical elevation of the SEM, $\alpha=\pi/4$

iv) $$R_{mag} = \begin{bmatrix} 1 & 0 & 0 \\ 0 & r & 0 \\ 0 & 0 & 1 \end{bmatrix}$$

scaling the vertical magnification

v) $$R_{z2} = \begin{bmatrix} \cos\theta_2 & \sin\theta_2 & 0 \\ -\sin\theta_2 & \cos\theta_2 & 0 \\ 0 & 0 & 1 \end{bmatrix}$$

$$\mathbf{X}_{screen} = R_{z2} R_{mag} R_x R_y R_{z1} \mathbf{X}_{sample} \ . \qquad (1)$$

The angles θ_1 and θ_2 and the constant r must be determined prior to any further analysis. This calibration is done in the following way:

- We create a synthetic image of the calibration sample.
- Varying the unknown parameters; θ_1 , θ_2, and r we compute the rotation matrices. These rotation matrices are then applied to the synthetic images to obtain their projection on the screen coordinate system.
- Next, the synthetic images are compared with those from the SEM. By matching the relevant slopes and sizes in both images, we find the set of necessary parameters.
- The rotation of the sample around the STM tip is determined each time a new sample is loaded inside the high vacuum chamber.

2.2 Fabrication of The STM Tip

For accurate imaging of the surfaces with STM, one needs a very sharp tip. However, for our purposes, to avoid buckling when it is in contact with the sample, the tip must be blunted. We therefore prepared our own tips from tungsten (W) wires using electrochemical etching. This involves anodic dissolution of the metal electrode [3]. We place the tungsten in a 2M NaOH solution. The tungsten wire which will be etched acts as the anode. The counter electrode is a ring which surrounds the wire. When a DC voltage (3 V in our case) is applied to the anode, W dissolves into soluble tungstate (WO_4^{2-}) anions. As a result of this reaction, tungsten is continuously etched forming a somewhat blunt tip.

2.3 Large Deflection Beam Theory

For the case of a cantilever beam subject to an end load P, the governing differential equation is:

$$\frac{1}{\rho} = \frac{\frac{d^2 y}{d x^2}}{[1 + (\frac{d y}{d x})^2]^{\frac{3}{2}}} = \frac{P x}{E I} \quad (2)$$

Where ρ is the curvature of the beam, y is the out of plane deflection, x is the distance from the support, E is the elastic modulus and I is the moment of inertia of the beam. For a detailed explanation of the solution to the large deflection problem, see Frisch-Fay [4]. Note that due to the large deflection there is a sliding contact between the STM tip and the sample during loading. This is accounted for in the analysis of the data.

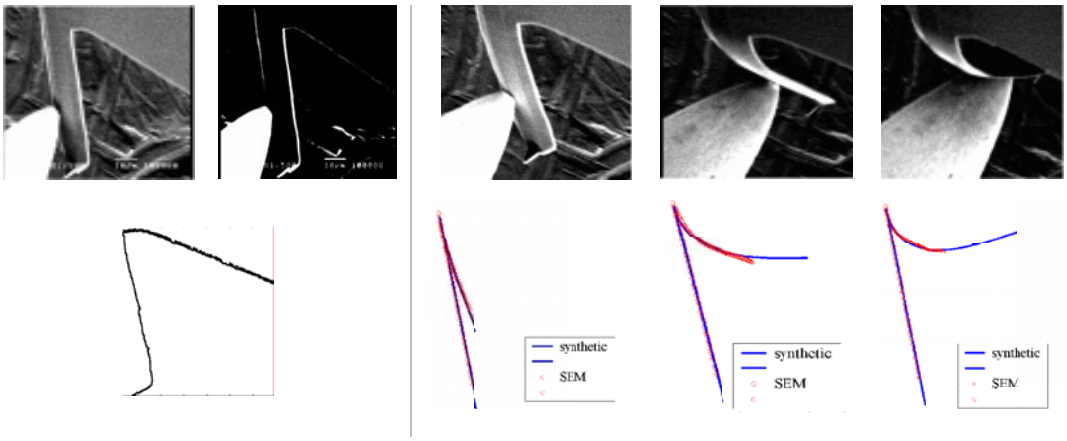
2.4 Digital Image Processing

To demonstrate the method, an experiment to test the fracture strength of commercially available 125 μm long silicon AFM cantilevers with a trapezoidal cross section was performed. To determine the fracture strength we first create a synthetic image having the same size and cross section as the tested cantilever. Using large deflection beam theory, the deflected shapes of this imaginary cantilever are obtained for increasing loads. These are then mapped onto the computer screen with the transformation matrices obtained through the above-mentioned calibration process. The next step is to compare these with the digital SEM images from the experiment, which correspond to different deflected shapes of the cantilever up to the point of fracture. The images of interest are filtered, their edges are detected and the right edge is plotted. The load applied on the imaginary beam is iterated until a good fit between the processed SEM and synthetic images is obtained, see Figure 4. By analysis of the last image obtained before fracture, the fracture load and hence fracture strength can be determined.

For the tested AFM cantilever we calculated the fracture strength to be 6.4 GPa. Next, we repeated the experiment with cantilevers annealed at different temperatures. The fracture strength after annealing showed variations from the untreated sample; we observed a fracture strength of 9.5 GPa after 375^{o}C, 6.5 GPa after 650^{o}C, 11 GPa after 825^{o}C, and 7.8 GPa after 1000^{o}C. Although we are not yet able to draw any conclusions because we don't yet have a statistical interpretation, we believe that this variation in fracture strength is related to the change in surface properties with annealing. SEM images of fracture surfaces (top views of the beams) of beams heated treated to different temperatures are presented in Figure 5.

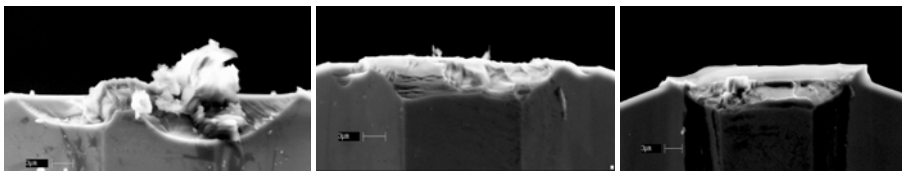
3 SUMMARY

We presented a new method to test the bending fracture strength of micron size scale cantilever beams. The new method uses an ultra high vacuum STM/SEM combination and is based on digital image processing. As a demonstration of the method we performed several initial tests to determine the fracture strength of Si AFM cantilevers with and without heat treatment.



a) b)

Figure 4 a) Edge detection b) Sequence of actual (top) and processed (bottom) images during fracture test of silicon beam (AFM cantilever).



a) b) c)

Figure 5: Fracture surfaces of samples tested after a) no treatment b) 825°C annealing c) 1100°C annealing. Scale bar on images is 3 µm.

4 ACKNOWLEDGEMENT

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5 REFERENCES

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