

Determination of Strength Degradation Mechanisms of Native Oxide on Silicon Nanostructures

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Abstract:

Understanding what affects the strength of micro- and nanoelectromechanical systems (MEMS and NEMS) is essential to effectively designing load bearing MEMS / NEMS. Research by Alan, et al. [1], suggests that oxide growth on silicon nanostructures leads to a decrease in strength, therefore increasing the likelihood of failure [1]. To interpret how silicon strength is affected as native oxide grows on the beams, the beams were failure tested weekly for five weeks allowing continuous growth of oxide, after which strength had decreased to 81% of the original strength. After the oxide was removed, strength recovered to 96% of the original strength. Silicon beam failure tests were performed using an atomic force microscope (AFM), which recorded the deflection of the AFM cantilever and the displacement of the piezo. A reference cantilever was used to find the stiffness of the AFM cantilevers used for failure tests. The beam's failure stress was calculated from the beams' failure force using the finite element method.

Introduction:

Due to factors such as large surface to volume ratio and low defect density, nanoscale strength cannot be determined using macroscale experiments. Silicon is a brittle material that fails stochastically, which is why many beams must be tested to obtain accurate strength data. Research suggests that uneven oxide layer growth may form a rough surface on silicon beams, thus causing the AFM cantilever forces to concentrate at one

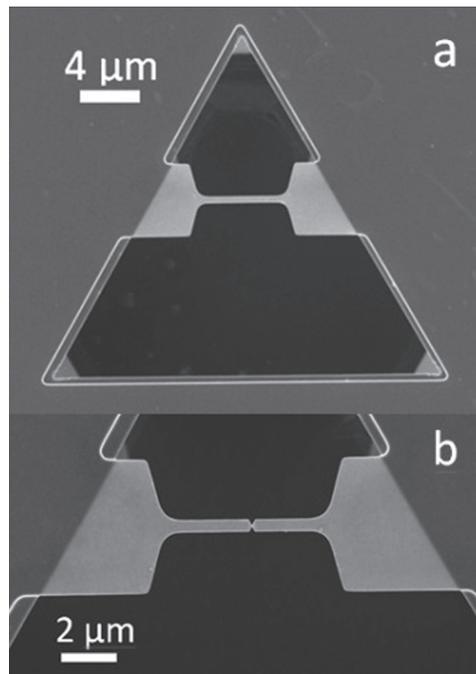


Figure 1: a) Top view scanning electron microscope image of beam and triangular trench. b) Beam with fracture at center.

of these stress points [1]. If roughness occurs, the beams' strength before and after oxide removal would be similar. Furthermore, if the strength is changed due to chemical interaction between oxide and silicon, the strength should increase after removal. To find how the mechanisms of native oxide growth correlate to silicon strength, nanoscale fracture specimens were fabricated and allowed to oxidize while tracking the reduction in strength. At the end, the oxide was removed and a final measurement of strength was made. The fabrication and strength analysis of the beams using an AFM was performed as described in Alan, et al. [2], and Namazu, et al. [3].

Methods:

Silicon $\langle 111 \rangle$ orientated wafers were used to fabricate the single crystal silicon beams. A photolithography step outlined the shape, and the beam thickness was etched using reactive ion etching (RIE). An oxide coat was grown on the wafer, and a second photolithography and RIE step defined the depth of the beam trenches. An anisotropic potassium hydroxide/ tetramethylammonium hydroxide etch released the beams by etching horizontally beneath them and terminating on their $\langle 111 \rangle$ orientation bottom surface. The beams were immersed into a buffer oxide etch removes the oxide to start performing the failure strength tests. The final beam sample is shown in Figure 1. To control the effect of humidity on rate of oxidation, the beams were stored at 100% relative humidity between strength tests.

The failure strength was measured using the deflection of the AFM cantilever and displacement of the piezo. The interaction between the piezo, AFM cantilever, and the beam were taken as springs in series (Figure 2); the piezo displacement equals the sum of the beam and AFM cantilever deflection [4]. A reference cantilever with a known stiffness was used to calculate the force applied on the beams. Finite element analysis was used to calculate the failure stress from the failure force (Figure 3). The failure stresses for each week fit a Weibull distribution as a group, which was used to measure average strength and plot as a function of time.

Results and Conclusions:

Testing in weeks two and three did not reflect the strength decrease as shown in Alan, et al. The beam strength, however, did increase surprisingly after oxide removal and resembled the strength prior to oxidation. Previous tests show that oxide reduces silicon beam's strength, which is not reflected from weeks two, three, and four (Figure 4). Our hypothesis about the cause of this discrepancy is the repetitive use of the same AFM cantilever. Since the same AFM cantilever was used for weeks one through four, debris from numerous beam fractures may have accumulated and caused this error in the data.

If only the data taken with new AFM cantilevers (weeks one, five, and six) are considered, then the results would fully reflect the degradation and restoration of beam strength. Techniques to protect silicon nanostructures from oxidizing and prevent strength reduction must be developed. Further analysis on the repetitive use of the AFM cantilever is needed to understand if this caused the error in the first few weeks.

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References:

- [1] T. Alan, A. T. Zehnder, D. Sengupta, and M. A. Hines. Methyl monolayers improve the fracture strength and durability of silicon nanobeams. *App.Phys.Letters*, 89:231905, 2006.
- [2] T. Alan, M. A. Hines, and A. T. Zehnder. Effect of surface morphology on the fracture strength of silicon nanobeams. *Applied Physics Letters*, 89:091901, 2006.
- [3] T. Namazu, Y. Isono, and T. Tanaka. Evaluation of Size Effect on Mechanical Properties of Single Crystal Silicon by Nanoscale Bending Test Using AFM. *Journal of Microelectromechanical Systems*, 9(4):450-459, 2000.
- [4] S. Sundararajan and B. Bhushan. Development of AFM-Based Techniques to Measure Mechanical Properties of Nanoscale Structures. *Sensors and Actuators*, 101:338-351, 2002.

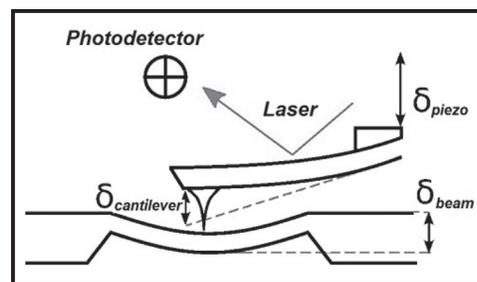


Figure 2: Side view schematic of the AFM cantilever and beam deflections.

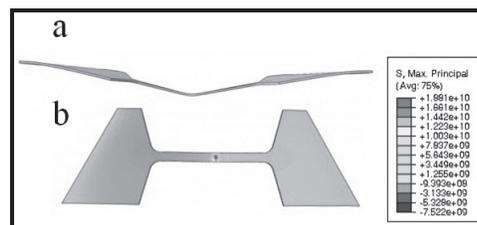


Figure 3: a) Side view finite element model of deflected beam. b) Top view of stress simulation. (See full-color version on inside cover.)

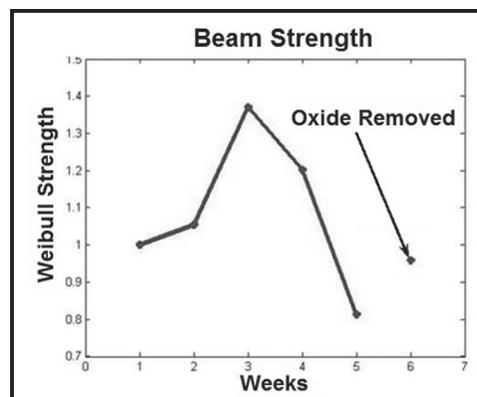


Figure 4: Normalized beam strength and weeks.