Use of MEMS Cantilevers to Characterize Young's Modulus in Silicon-Rich Silicon Nitride

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***Abstract*— In this paper we develop sample MEMS devices, using cantilever and fixed-fixed beams from a silicon-rich silicon nitride film, and test them to determine physical properties of silicon nitride. Our data from the devices appears consistent with other literature, showing a Young's modulus of approximately 200 GPa, and a residual stress that can be on the order of 20-90 MPa. Analysis of this data confirms the strong dependence of bending stiffness, used to determine the modulus and residual stress, on the geometries of the beams, and the importance of precisely measuring the critical features to produce accurate data.**

I. INTRODUCTION

WITH the increasing research into microelectromechanical systems (MEMS), physical properties of silicon and related materials have become as important as the previously relied upon electrical properties. To develop mechanical systems on such a small scale, it is important to know how materials will react to the forces put upon them. Applications, such as MEMS used in atomic force microscopes, rely upon exact knowledge of these properties to function as desired.

In this work, we develop and analyze properties of MEMS cantilevers and fixed-fixed beams formed from silicon nitride film on silicon substrate. By applying forces to these devices, we can measure the deflection, giving data from which we can calculate Young's modulus and residual stresses in the silicon nitride. These values are particularly important in developing MEMS devices, as Young's modulus determines how a material deforms with applied stress, and the residual stress determines how much additional stress can be applied to a device before it will yield or permanently deform.

II. EXPERIMENT

To begin the fabrication process, a 1μm layer of silicon-rich silicon nitride was grown in LPCVD. This layer was then measured to determine its thickness and refractive index. The refractive index was used to extract the ratio of silicon to nitrogen in the silicon nitride layer, which determines the physical properties it exhibits. The thickness was measured to be 1.02μm, very close to our expected growth, and the refractive index of 2.3, indicating a silicon to nitrogen ration between 4 and 6 [5]. Such a high ratio will cause the silicon nitride to have a thermal expansion coefficient closer to that of bulk silicon, reducing the residual stress caused by the differing contractions when the wafer cool.

After characterizing the silicon nitride layer, the wafer was prepared with a photoresist, and then exposed through contact lithography. Our masks create patterns for cantilevers and fixed-fixed beams in an array of lengths and widths. Once exposed, the photoresist was then developed to harden, preparing the wafer for etching. Etching was done by SF_6 plasma, which reacts chemically with the silicon nitride to remove the areas revealed through the exposed photoresist. The photoresist was removed, and the finished result was inspected under an optical microscope to ensure that the wafer was fully etched. Measurements were also taken to find the new film thickness of the silicon nitride, measured at 0.99μm. The change in thickness is quite small, due to the protective layer of photoresist.

Once etched, the wafer was placed in a KOH bath. KOH was chosen for its selectivity, etching much faster in the $<100>$ and $<110>$ planes than in the $<111>$ plane of silicon. This selectivity creates an undercut, etching away the silicon beneath the silicon nitride cantilevers. After this final etch, wafers were inspected again under an optical microscope, making sure that the silicon beneath the cantilevers was fully removed.

The fully etched wafers were then tested using a nano-indenter, taking data on loading versus deflection for various sizes of cantilevers and fixed-fixed beams. This data was then processed to extract values for Young's modulus and residual stress of silicon nitride.

III. RESULTS & DISCUSSION

Comparing the results from our data on the Young's modulus and residual stress in silicon nitride, we see that our cantilevers compared closely to our expectations, based upon moduli found in literature [2]. Our fixed-fixed beams, however, had large deviations from the expected moduli. These deviations can be attributed to various errors and tolerances in our measurements. The fixed-fixed beams do have residual stresses consistent with those found by Sekimoto [5], when we use the appropriate modulus that we found from the cantilevers.

When analyzing the cantilever beams, we expect a linear relationship between the applied force and the deflection of the beam. Our data, shown in Figure 1 and 2, shows this relationship for both the 50μm $\times$ 100μm and the 50μm $\times$ 50μm cantilever, as well as the linear fit that approximates the data. Based upon the sizes of these cantilevers, using the 1μm thickness that we measured earlier, we extracted a Young's modulus of 216GPa for the 100μm long cantilever, and a modulus of 210GPa for the 50μm one. These are very similar to the results found by Guo, who quotes a modulus of 195 $\pm$ 9GPa [2].

At this point it is important to notice how sensitive the modulus is to the geometry of the cantilevers. The modulus

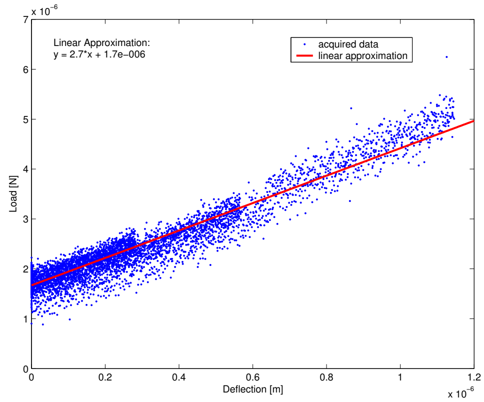

Fig. 1. Beam bending in a 50μm $\times$ 100μm cantilever.

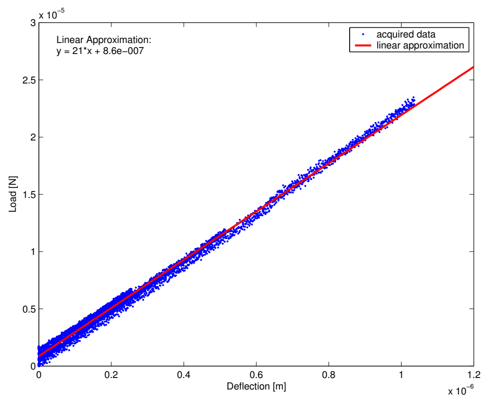

Fig. 2. Beam bending in a 50μm $\times$ 50μm cantilever.

is proportional to the ratio of length to height raised to the third power. This means that a 10% error in one of these measurements can result in a 33% error in the calculation of the modulus. This seems particularly important when we consider that the nano-indenter has a tip radius of 10μm, and cannot rest exactly at the tip of the cantilever without slipping off when it the cantilever bends. To compensate, we can consider the cantilever's length to be slightly less than it's actual length, as the force is being applied to a point inward of the tip. This inward distance was on the order of 5μm, but could not be accurately measured, introducing error into the calculations. In the previous calculations, we used lengths of 95μm and 45μm for the 100μm and 50μm cantilevers, respectively. The uncertainty in the exact length, however, must be noted when considering the exactness of the modulus.

The third cantilever measured bore much poorer results than the initial two, but deserves investigation. The beam measured 50μm $\times$ 500μm, a ratio of 10:1 much closer to classical studies of beam bending than the shorter beams we measured previously. However, the data, shown in Figure 3, does not correlate nearly as well as the shorter, stiffer beams. With such a long beam, it small applied forces can create a great deal of deflection, such that the nano-indenter cannot resolve the forces before reaching its maximum deflection. The indenter attempts to find the surface of the cantilever by lowering its tip until it measures a 2μN force. In most cases,

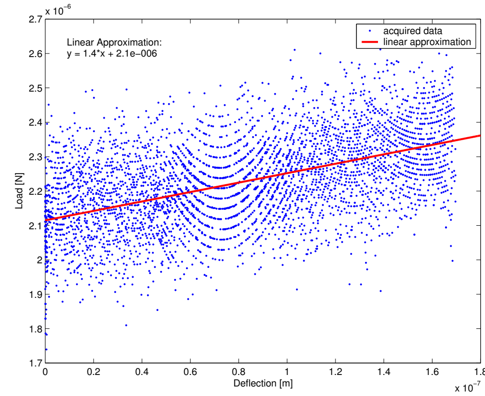

Fig. 3. Beam bending in a 50μm $\times$ 500μm cantilever.

the deflection required to cause this is negligible, but with this long cantilever, the force does not reach 3μN before the indenter has reached its full extent. This produces a great deal of noisy data, with little correlation to Young's modulus.

One interesting feature to note in the longer cantilever is the very regular series of curves that develop in the seemingly noisy data. These curves are likely due to the cantilever resonating as it is depressed by the indenter tip. The oscillations produced are a function of the geometry and modulus of the cantilever, and are another means of determining the modulus of the silicon nitride film. A similar method using natural resonant frequencies was used by Guo in his determination of the modulus of silicon nitride [2].

While the cantilevers we manufactured provide a simple linear relationship between load and deflection, the fixed-fixed beams allow us to examine both modulus and residual stress in the silicon nitride. Unfortunately, the relationship is not linear, but contains a third order term dependent on Young's modulus. By matching this third order term, we can determine the modulus, and use it to find the residual stress. From the data in Figures 4 and 5, we calculate a modulus of 45GPa for the 5μm $\times$ 50μm beam, and a modulus of 49GPa for the 10μm $\times$ 50μm beam. These values are quite low when compared to the expected 195GPa.

One possible explanation for this discrepancy comes from the geometry of the beams. Since the beams are not perpendicular at their fixed edges, the boundary conditions may be changed slightly. Also, if the tip of the indenter were not exactly at the center of the beam, the deflection would not have been even on both sides. Both these effects would change the effective length of the beams. As with the cantilevers, the modulus is proportional to the cube of the length, so that any change in length greatly influences the calculated modulus. However, to account for the difference in the calculated modulus, the effective length would have to be 60% longer than its measured length. This seems unlikely, particularly given that placement of the indenter tip away from the center of the beam would decrease the effective length, making the beam stiffer and the modulus higher. It is also unlikely due to the fact that both measurements of the modulus are consistent with each other, while the random error in centering the indenter tip would imply less correlation between trials.

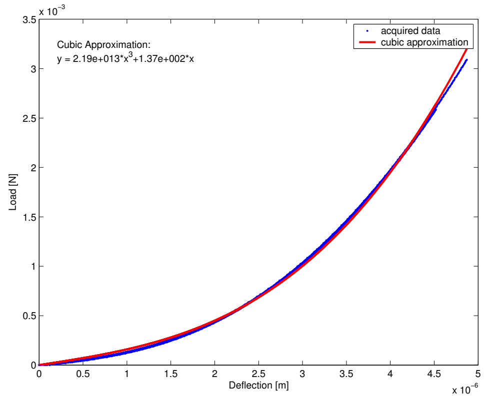

Fig. 4. Beam bending in a 5μm $\times$ 50μm fixed-fixed beam.

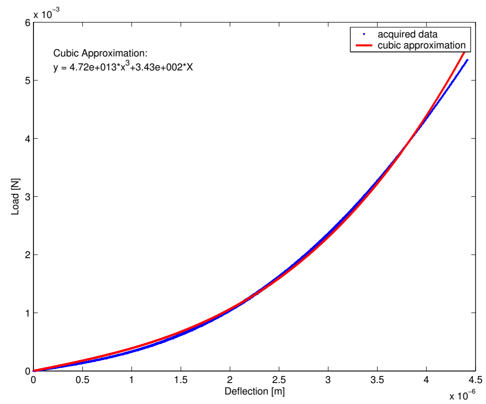

Fig. 5. Beam bending in a 10μm $\times$ 50μm fixed-fixed beam.

Another possible explanation is that, as it is deflected further, the beam begins to undergo plastic as well as elastic deformation. When the deformations become non-reversible, in the plastic regime, our equations of deflection become less accurate. The plastic deformation will cause the beam to deflect more than we expect it to, resulting in what looks like a lower modulus.

If we assume a more standard Young's modulus than what we calculated from our fixed-fixed beams, we can examine the linear region, at small deflections, to determine the residual stress within the beams. This stress, calculated as 14MPa in the 5μm $\times$ 50μm beam and 84MPa in the 10μm $\times$ 50μm beam, is in rough agreement with Sekimoto's findings of residual stress ranging from 10-70MPa. If the residual stress becomes large enough, it starts to dominate the linear regime of the fixed-fixed beam bending, causing deflection to deviate from our predictions. However, for the 5μm $\times$ 50μm beam, we can safely ignore the effect of residual stresses less than 200MPa, and can ignore residual stresses in the 10μm $\times$ 50μm beams for values less than 3.1GPa. As silicon nitride has a yield stress on the order of 200MPa, residual stresses at these values would cause the fixed-fixed beams to fail, so this also puts a limit on the magnitude of acceptable residuals stresses. This factor drove the choice in using silicon-rich silicon nitride, as it has much lower residual stresses than standard stoichiometric silicon nitride.

IV. Conclusion

In this paper, we have shown how cantilever and beam bending can be used to determine Young's modulus and residual stress in materials for MEMS technologies. However, the oversensitivity to errors in measuring the exact geometries of the features leads to an uncertainty that is higher than acceptable for anything beyond a first order approximation. To achieve a higher precision measurement of the modulus, more complex methods, such as resonance analysis, are required. For our purposes, these results show a close agreement with the existing literature, providing a simple method of determining modulus with linear and cubic models.

Acknowledgment

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APPENDIX I
DATA

APPENDIX II
CALCULATIONS