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Evaluation Of Elastic Modulus And Stress Gradient Of PECVD Silicon Nitride Thin Films

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ABSTRACT: This study investigated the techniques for determining the elastic modulus and estimating the stress gradient of plasma-enhanced chemical vapor deposition (PECVD) silicon nitride thin films. The experimentally determined elastic modulus was then used in a finite element beam model to compute the stress distribution inside the thin films using a commercial finite element analysis package. The computed beam displacement caused by a given stress gradient was compared with the displacement experimentally evaluated using optical interference microscopy. This comparison allows the stress gradient of the PECVD silicon nitride membrane introduced by the fabrication process to be evaluated.

1. INTRODUCTION

In recent years PECVD silicon nitride (SiN_x) has found increasing application as a structural material in microelectromechanical systems (MEMS), such as static membranes, tunable inductors and tunable MEMS Fabry-Pérot (FP) optical filters [Dell et al., 2002]. The advantage of PECVD SiN_x is that it can be deposited at much lower temperatures than SiN_x deposited by the more commonly used low-pressure chemical vapor deposition (LPCVD) method [Winchester and Dell, 2001]. This is beneficial in fabricating devices that are temperature sensitive. However, evaluation and control of mechanical properties, such as elastic modulus and intrinsic stress, in thin films are critical to achieve required device performance and yield.

SiN_x thin films are used to construct tunable FP optical filters in this study. As illustrated in Figure 1(a), the PECVD SiN_x thin film forms a membrane structure that is suspended on four pillar supporters via suspension arms. The membrane carries a mirror on its top surface, moving up and down under electrostatic force in response to a bias voltage applied between the membrane and the ground. Mechanical characteristics of the silicon nitride membrane, including the elastic modulus of the material, the intrinsic stress gradient through the thickness of the SiN_x layer and the mechanical design of the structure, are thus of great significance in determining structural integrity and filter performance. Figure 1(b) shows the variation in the modeled response of the cross membrane structure in Figure 1(a) with the elastic modulus as a parameter. A change of modulus value from 220 GPa, a typical value for bulk silicon nitride, to 150 GPa can result in a membrane displacement variation of ~50 nm at the applied bias of 1 V. The variation is significant when constructing a FP filter that is to operate over a specific spectral range. Of the same importance is the degree of any intrinsic stress gradient existing in the SiN_x layer, which causes inherent deformation to the membrane and thus affects the optical performance of the filter. Therefore, it is essential to understand the properties of low-temperature deposited PECVD SiN_x films if this material is to be successfully applied to MEMS devices.

This paper reports on a nanoindentation investigation to determine the elastic modulus of PECVD SiN_x thin films. The experimentally determined modulus was then used in a finite element cantilever beam model to estimate the intrinsic stress gradient resident in fabricated PECVD SiN_x beams. By comparing the modeled beam deflection with the experimentally measured beam deflection, the stress gradient was then evaluated. Further verification of this method was carried out in a cross membrane structure.

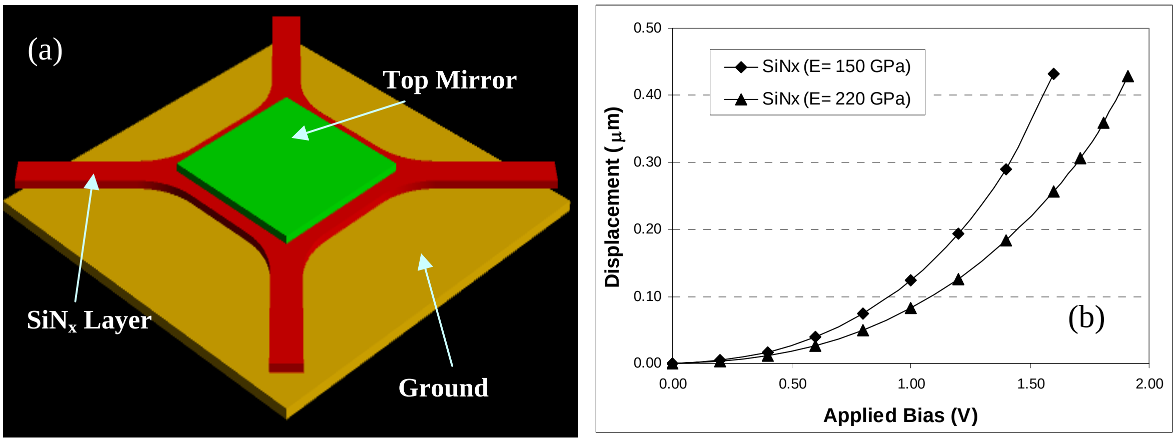

Figure 1: (a) The FP filter consists of a top mirror, a SiN$_x$ layer and a ground mirror. The SiN$_x$ layer has a square membrane (with dimensions: 100 x 100 x 0.4 μm) supported at each corner by four arms (with dimensions: 100 x 20 x 0.4 μm). There is a cavity (1.2 μm in length) between the membrane and the ground. (b) Boundary and finite element modeled membrane deflection is plotted as a function of applied voltage.

2. EXPERIMENT

SiN$_x$ thin films were deposited by PECVD at 125°C using $SiH_4/NH_3/N_2$ precursors. The SiN$_x$ films for nanoindentation measurement were deposited on 300 μm thick silicon (Si) substrates. The film thicknesses were 350, 390 and 700 nm. The films were amorphous with a small volume fraction of porosity. Atomic force microscopy (AFM) showed smooth film surfaces with roughness less than 10 nm. For micro-cantilever beams fabrication, the SiN$_x$ was deposited onto polyimide using the identical conditions to those made for nanoindentation, and then released in oxygen plasma. The beam deflection was measured using an optical microscope fitted with a green interference filter [Martyniuk et al. 2004].

Nanoindentation experiments were conducted using a HYSITRON TriboScope nanomechanical testing instrument. A Berkovich indenter with a tip radius of 100 nm was used. For each specimen, 64 indents were performed with a load ranging from 50 μN to 9000 μN. Determination of elastic modulus was made from the initial part of the unloading-displacement curves [Oliver and Pharr, 2004], over indenter penetration depths ranging from 20 nm to 200 nm. AFM was also used to confirm that the indents were well-formed over this penetration range. The elasticity modulus of the bilayer specimen was calculated by using Equation 1.

$$\frac{1}{E_{eff}} = \frac{1-\nu_i^2}{E_i} + \frac{1-\nu^2}{E} \qquad (1)$$

where E_{eff} is the effective modulus determined from the load-displacement curve, E_i and ν_i are the modulus and Poisson's ratio of the diamond indenter, and E and ν are the modulus and Poisson's ratio of the specimen. Values of E_i = 1141 GPa, ν_i = 0.07 and ν = 0.27 were used in this study.

Finite element modeling of stress gradient was conducted using commercial software CoventorWare™ Version 2003.1. For cantilever beam models, all the nodes at the fixed end of a beam were constrained in the directions of x, y and z axes. The cantilever beam models were meshed using the 27-node Manhattan Bricks. The cross membrane model used 1883 27-node Extruded Bricks, with the same boundary condition of no displacement along x, y and z axes applied to all the nodes at the fixed end of the arms.

3. RESULTS AND DISCUSSION

3.1 Nanoindentation

Figure 2 shows the nanoindentation measurements of elastic modulus of the PECVD SiN_x/Si bilayer specimens as a function of the indent contact depth. Also shown in the figure is the measurement of the elastic modulus of the Si substrate. It is seen that the measured moduli of all three specimens are less than that of the Si substrate and increased asymptotically to that of the Si substrate with increasing contact depth *h*. It is expected that for bilayer systems the measured modulus via indentation should eventually reach that of the substrate [Bhattacharya and Nix, 1988, Jung et al., 2004], which is 168 GPa in this case. However, the maximum load of the indenter used in our experiments is limited to 9000 μN, which is insufficient to see saturation of the modulus value.

It is commonly believed that for a film/substrate bilayer system the elastic modulus of the film can be measured only if the ratio of indentation depth to film thickness is below a critical value, e.g. 0.1. For this case, the data obtained at smaller contact depths, e.g. when $h < 50$ nm, appeared to be not reliable, mainly due to the size effect of the indentor tip [Wei et al. 2004], which had a nominal radius of 100 nm in this case. As shown in Figure 2, there appeared to be a plateau in the modulus value for each specimen when *h* is within the range of 50 to 80 nm, indicating that within this range of contact depth the effect of substrate on the measured modulus could be negligible. The plateau values are considered the true values of elastic modulus of the thin films. The determined modulus values for the films of 390 and 700 nm in thickness were ~100 GPa. The modulus for the film of 350 nm was measured to be 108 GPa, slightly higher than the values for the two thicker films. The result indicated that for the SiN_x/Si bilayer systems studied here the minimum film thickness for obtaining reliable film modulus was about 400 nm. This further suggested that a thicker film can provide more reliable estimation of the true film property.

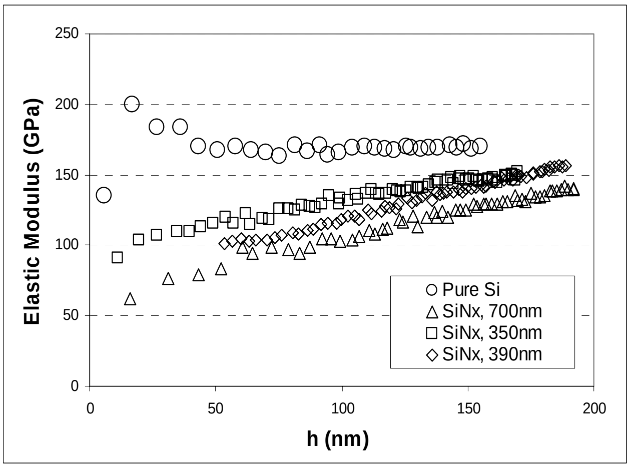

Figure 2: Elastic moduli of SiN_x/Si bilayer systems and Si substrate are plotted as a function of indenter contact depth, *h*.

3.2 Evaluation of intrinsic stress gradient

Intrinsic stresses can be easily generated during processing of low-temperature deposited SiN_x films. The evaluation of intrinsic stresses in thin films is essential in order to realize the required performance and reliability of any MEMS device where thin films are used. A typical method to assess the internal stresses is to measure the stress-induced substrate curvature using an optical setup. From the measured curvature, the average stress in a thin film can be estimated using the Stoney formula [Stoney, 1909]. X-ray diffraction (XRD) methods have also been utilized to measure the stress residing in thin films [Zhang et al., 1999]. In this method, the shift of the {100} crystal plane diffraction peak of the substrate caused by internal strain is detected by XRD. The stress was then calculated by multiplying the strain with the elastic modulus. The method is effective only if stress in thin films is sufficiently high that the induced substrate strain can be detected by XRD. The estimated stress is that in the near interface layer between the film and the substrate.

To date, there does not appear to have been reported any reliable methods that are able to measure stress gradient through thickness (i.e. along z-axis) in thin films. In this study, a method that utilizes both experimental and numerical results for evaluating the stress gradient in SiN_x films was investigated. In this method, a cantilever beam of 200 μm in length, 20 μm in width and 500 nm in thickness was fabricated using low-temperature PECVD of the SiN_x film followed by oxygen plasma sacrificial layer removal. Upon the release, the beam was deflected under the influence of the internal stress gradient in the thin film. The beam profile was measured along the length using an interference microscope, with the results shown in Figure 3(a) as solid symbols. A finite element model of the cantilever beam was then built. With the assumption that the stress gradient was the dominant factor to cause the beam deflection, the beam profiles were numerically obtained by employing different values of linear plane stress gradient along the z-axis. The best fit of the computed beam profile to the measured data gave an estimate of the stress gradient in this thin film. The computed beam profile under a stress gradient of 275 MPa/μm was found to be the best estimation, which was also plotted in Figure 3(a). To further test the reliability of this method, cantilever beams with different lengths were fabricated using the same processing conditions as those used to obtain the data in Figure 3(a). Their corresponding FEM models were also built. This time, the stress gradient of 275 MPa/μm estimated earlier was used in all the models and the beam deflections were thus computed. The comparison between the measured and computed tip deflections, shown in Figure 3(b), indicates a good agreement between the finite element modeling and the experimental data. Minor differences could be due to the assumption of a linear distribution of the stress gradient in the films and the beam dimensional error from the fabrication.

The method was also used to evaluate the stress gradient in a Fabry-Perot SiN_x membrane structure fabricated using the low-temperature PECVD SiN_x and an oxygen plasma release process. The stress gradient value of 275 MPa/μm was again used in the cross membrane FEM model to compute its resultant deformation. As shown in Figure 4(a), the computed deformation along z-axis at the center of the membrane is -469 nm (i.e. bowing downwards) and the deformation along the edge of the membrane is +532 nm (i.e. extending upwards). The PECVD fabricated cross membrane structure, that has the same dimensions as those in Figure 4(a), has a deformation of about -500 nm at the center and a deformation of about 500 nm along the edge, estimated using its interference pattern shown in Figure 4(b). The deformation values determined experimentally and numerically are in reasonably good agreement. Careful comparison of the two images in Figure 5 shows that their deformation patterns are rather similar. The differences in deformation values could be attributed to the complexity of the cross membrane structure being modeled, as well as the dimensional inaccuracy caused by the fabrication. The MEMS fabrication might produce a dimensional inaccuracy of several percent and a certain level of non-uniformity in membrane thickness, which was not considered in the finite element model. In future studies the FFM model will be refined to allow improved stress gradient evaluation.

4. CONCLUSIONS

The nanoindentation technique was found to be an effective method to measure elastic modulus of low-temperature deposited PECVD SiN_x thin films. The accuracy of this method relies on the proper interpretation to indentation results. This study has demonstrated that the effect of the substrate must be removed to obtain a reliable modulus values for the thin films considered here.

By comparing cantilever beam deformations predicted by finite element modeling with the experimentally determined beam deflection results, the intrinsic stress gradient in the thin films can be estimated. Experimental verification with the cross membrane structure indicates that this method is reliable. However, model refinement is needed to achieve a more accurate estimation of stress gradient.

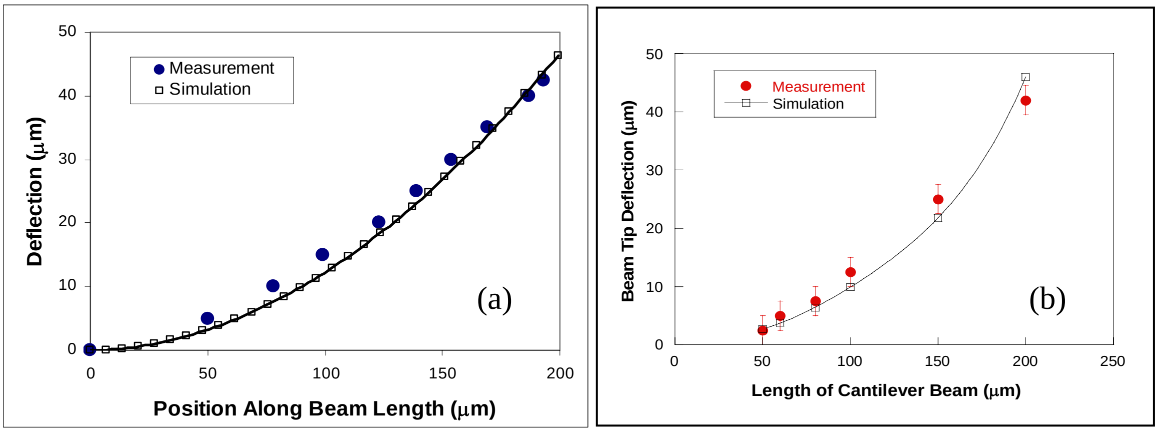

Figure 3: (a) Measured and computed deflection profile of a cantilever beam. Beam is 200 μm in length, 20 μm in width and 300 nm in thickness. (b) Comparison between measured and computed tip deflections of cantilever beams with various lengths under a stress gradient of 275 MPa/μm. E = 100 GPa was used in the modelling.

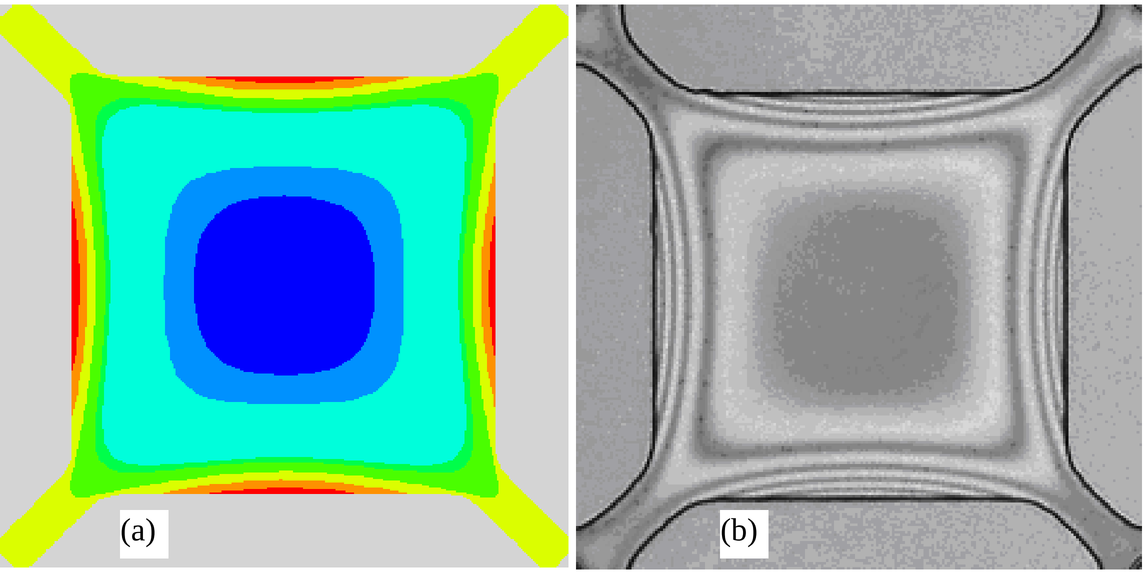

Figure 4: (a) Deformation contour map of the modelled membrane structure was obtained under a stress gradient of 275 MPa/μm. E = 100 GPa was used in the modelling. The square membrane has an edge length of 100 μm and a thickness of 400 nm which is supported at its corners by four arms. The arm dimensions are 20 μm in length and 10 μm in width. (b) PECVD fabricated cross membrane structure using deposition temperature of 125 °C in a $SiH_4/NH_3/N_2$ atmosphere followed by the oxygen plasma releasing. Each interference fringe indicates a deformation of 250 nm.

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