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Measurement of Mechanical Properties of Low Stress Silicon Nitride

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Abstract

Low Stress Silicon nitride beams were fabricated to determine Young's modulus and residual stress of the thin film. The structures were mechanically loaded and the corresponding deflection was measured. From the load versus deflection curves, an effective spring constant was calculated. The spring constant was used to analytically determine a fundamental material property, the Youngs Modulus, of the silicon nitride. A decrement in the Youngs Modulus of the cantilever was observed and the percentage error found was approximately forty-four percentage. The value of the Residual Stress of thin films decreased from the expected value and the deviation was estimated to be twenty-two percent.

Index Term-Youngs Modulus, Residual Stress, Silicon Nitride thin films.

I. INTRODUCTION

The evaluation of the mechanical properties of thin films is indispensable for designing Microsystems (MEMS)devices, since the properties are closely connected to the device performance. Elastic properties, such as Youngs Modulus, Poisson's ratio and shear modulus etc. are related to the functionality of the micro systems. The Youngs Modulus of the thin films is proportional to the stiffness of a device structure.

During the experiment Youngs Modulus and the Residual Stress of the low stress silicon nitride structures were determined. The structures were mechanically loaded and the corresponding deflection was measured. From the load versus deflection curves, an effective spring constant was calculated which is used to determine the Young's Modulus of the silicon nitride. The cantilever showed linear elastic behavior and the fixed -fixed beam varied in non-linear fashion. The Youngs Modulus determined was 146.42GPa and the residual stress was calculated to be0.179 GPa.

Analysis of these mechanical properties of the thin film of silicon nitride was done using a HysitronTriboIndenter.

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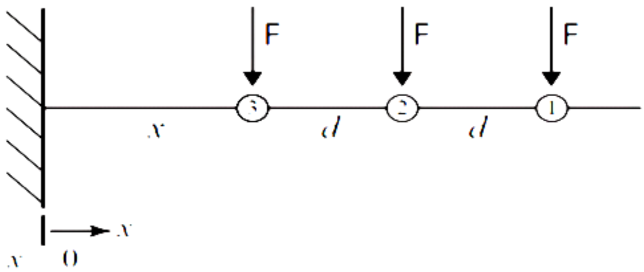

Fig.1. Force*F* being applied to the cantilever at three precisely separated locations for measuring the of the force constant *K* .

II. EXPERIMENT

II.a. FABRICATION PROCESS

The wafer was treated with Hexamethyldisilazane (HMDS) vapor followed by deposition of 4micron thick film of positive photoresist using the spin-on process. A hot plate at 90°C wasused for the soft baking of the wafer. Then the wafer was exposed to Ultra-Violet light for 5 seconds using a mask aligner and subsequently post baking is done at 130°C for 5minutes. A microscope was used to inspect the wafers and for assessment of the overall result of photolithography. Dry-Etch of Silicon Nitride was done in plasma and Sulfur Hexafluoride. The photoresist was removed in the asher with the ashing time of 8 minutes per wafer. Silicon is etched in wet Potassium Hydroxide (KOH) bath at 80°C. Anisotropic etching of Silicon is done by addition of 20% KOH in 3 liters of (DI) Deionized Water. After KOH wet etching for 1-2 hours the wafer was rinsed again with Deionized Water (DI) water and sprayed with isopropanol. Isopropanol spray is used to avoid the stiction of the released cantilevers to the<111> side walls. The wafer was dried and the etching results were inspected under a microscope. A Diamond scribe was used to cut the wafer into individual groups, each of four dies.

II.b. TESTINGBY NANOINDENTATION

The cantilever and fixed-fixed beams were mechanically tested to determine Youngs Modulus and the Residual Stress of the material. The structures were mechanically loaded and the corresponding deflection was measured. From the load versus deflection curves, an effective spring constant (k) was calculated as in Fig. (3) The spring constant was used to analytically determine a fundamental material property, the Young's Modulus, of the silicon nitride thin film.

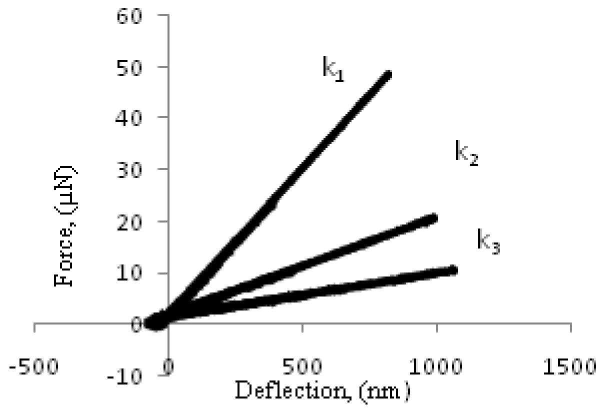

Fig.2. Force constants k_1=0.0571, k_2=0.0192, and k_3 =0.0082 is calculated by plotting Forceversus Deflection.

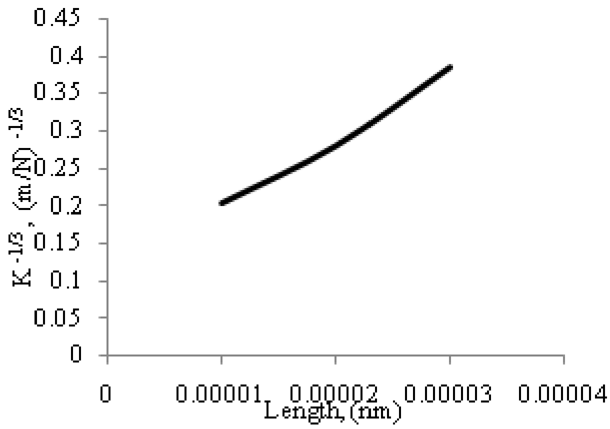

Fig.3. Graph is plotted between $K^{-1/3}$ and *L,* the gradient of the line is used to determine the Youngs Modulus by using$b$= $(3EI)^{-1/3}$. The Youngs Modulus is calculated to be 106.421GPa.

The residual stress was calculated by plotting a graph between F/z and z, where F is the normal force and z is the deflection. Gradient of the straight line is influenced by the Youngs Modulus of the material while the Y- intercept gives information about the geometry and the residual stress of the structures.

III. RESULTS AND DISCUSSIONS

The data from one force– deflection cyclewasconsideredforthe determination of the Youngs Modulus. The gradient of a *F–z* graphwasassumed to be equalto $3EI/L^3$ fora long narrow beam, where *F* is the normal force acting on the beam,*z* is the deflection, *E* is the Youngs Modulus of the Silicon Nitride thin films and *L* is the length of the beam, *I* is the moment of inertia of the material, estimated by using $3EI/\ [(1-v^2)L^3]$

,for a plate and by eliminating the offset fromtheforce measurements. Thismethodwasstraightforward, butbecause the position of theNano indenter tip relative to the end of the beam was known to be withinno fewer than afew micrometers,the estimate of*E*was a subject to substantialuncertainty.

A refined extraction approach wasused to determine the spring constant$k = F/z$ of a beam at each of the three locations where the nanoindentertip made contact with the beam. Graph wasplotted between $k^{-1/3}$and L, the nominal distance from the cantilever's root to the location of the Nanoindenter tip. The gradient of the ($k^{-1/3}$)–L graphwas assumed to be equalto$(3EI)^{-1/3}$ fora beamor$[3EI/(1-v^2)]^{1/3}$ for a plate. Inthis way the uncertainty wasreducedwhile extracting the value of Young's modulus. Where$E$ is Young's modulus, v is Poisson's ratio and z isthe deflection, I is the moment of inertia$w$ is the width and $t$is the thickness.

$$I = wt^3/12, \quad (1)$$

$$z = FL^3/3EI, \quad \text{for a long narrow beam}, \quad (2)$$

$$z = (1 - v^2)FL^3/3E, \text{for a plate.} \quad (3)$$

One cantilever,50nm×100nmwas tested, with a load$F$ being applied to cantilever at three precisely separated locations.Force was applied at *10μm, 20 μm* and *30μm* away from the free end of the cantilever beam. The normal forces applied at three different points were*50μN, 100μN* and *200μN.* The force constant k was calculated by using $F=k\,x$, by plotting a graph of load versus displacement.The force constants k_1, k_2 and $k_3$were calculated for the cantileverbeam at the three different points of loading as shown in Fig. 2.The values of Force constants calculated isk_1=0.0571, k_2=0.0192, and k_3 =0.0082.Then graph was plotted against $(K)^{1/3}$ and x and the slope $b= (3EI)^{-1/3}$was determined, as shown in Fig. 3. The Youngs modulus was calculated by using the formula

$$E = \frac{4}{wt^3b^3} \quad (4)$$

The Youngs Modulus for the cantilever beam was calculated to be 146.421GPa.

One of the fixed-fixed beams, 10nm ×50nm,of length L, width w and thickness$t$, with a residual stress in the film of $\sigma_0$wasloaded with force F at its center. The width was measured to be 8.39μm, the length was 49.μm and the thickness was around 1μm.The fixed-fixed beam was deflected andis loaded until the beamwas fractured. The forces applied were 3000μN, 6000μN and 120000μN. The beam fractureoccurred at 9000μNforce. Whenagraph was plotted between F/z andz^2a straight line was formed whose gradient depends on E and F/z intercept depends on geometry, E and residual stress. By using equation (5) the Residual stress was calculated to be 0.179 GPa. The force at the center is given as:

$$F = \left\{\left(\frac{\pi^2}{2}\right)\left[\frac{\sigma_0 wt}{L}\right] + \left(\frac{\pi^4}{6}\right)\left[\frac{Ewt^3}{L^3}\right]\right\}z + \left(\frac{\pi^4}{8}\right)\left[\frac{Ewt}{L^3}\right]z^3 \quad (5)$$

It was noted that the nominal beam 'width' of 10 μm was measured parallel to the edge of the etch pit. The bridges were oriented at an angle of arc tan (4/3) to the edges of the etch pits, so that the actual transverse width of this bridge (as it appeared on the photolithographic mask) was around 8μm.Berkovich indentation test was performed on SiN_x/Si wafers after deposition but before patterning or etching. Nano indenter tip was pressed into the film and the gradient of the force–displacement trace was used to estimate the reduced modulus E_r, which is related to Young's modulus, E, of the material as follows:

$$\frac{1}{E_r} = \frac{(1-\nu^2)}{E} + \frac{(1-\nu_i^2)}{E_i} \quad (6)$$

Equation(6)ν is Poisson's ratio of the SiN, E_i is Young's modulus of the diamond indenter tip (1100 GPa), and ν_i is Poisson's ratio of diamond (0.07).The maximum depth of indentation was ~70 nm. The value of the reduced modulus, averaged over 15 locations on the film, was 171.25 GPa.

TABLE I THE YOUNGS MODULUS AND RESIDUAL STRESS VALUES WERE CALCULATED FOR THE 50nm×100nm CANTILEVER BEAM AND 10nm×50nm FIXED-FIXED BEAM RESPECTIVELY.

Results	Youngs Modulus	Residual Stress
Calculated Values	146.42GPa	0.179GPa
Expected Values	190±5GPa	0.342 GPa
% Error	44%	22%

In the tests the errors might have occurred due to the inaccuracy in determination of the beam and the test geometry. Some drawbacks would have arisen due to effect of the substrate properties of the Nano indenter during testing. The deviation in the value of Youngs Modulus was possibly due to the non-uniformity of the LPCVD (Low -Pressure Chemical Vapor Deposition) Silicon Nitride thin film, as was observed by the change of the reflected color across the wafer. The decrease in the Residual stress value was reported due to the high temperature deposition conditions. Thermal stress was produced due to the difference in the expansion coefficient of Silicon and Silicon Nitride, subsequently decreasing the Residual Stress of the Si on SiN_x. The film growth mechanism and the amorphous nature of the film might have also contributed to the variation in the values of the Youngs Modulus and the Residual Stress.

IV. CONCLUSION

The Youngs Modulus measurement of the low stress Silicon Nitride structures have been performed by plotting load versus deflection graph and was calculated to be 146.42 GPa. There was decrement in the YoungsModulus and the deviation was approximately 44%. Linear behavior of the force and the displacement curve was observed for the (50nm×100nm) cantilever and non-linear for the (10nm×100nm) fixed-fixed beams. The thin film's residual stress was determined as 0.179 GPa and the percentage error was estimated to be 22%.

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