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A Noninvasive, on Wafer Method to Determine the Intrinsic Stress of a Polymer Layer

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Abstract The method described in this paper allows an investigator to determine the intrinsic stress of a polymer layer in a way that does not result in damage to devices or test structures. The method requires that a small area of the polymer be released from the substrate to form a diaphragm. The diaphragm is stimulated with acoustic white noise and the diaphragm movement is monitored with a laser vibrometer. The first few resonance frequencies of the diaphragm are obtained using a laser vibrometer and then those frequencies are used to calculate the membrane intrinsic bi-axial tension.

Keywords Diaphragm · Testing · MEMS · Fabrication

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Introduction

Polymer materials are becoming more common in the fabrication of MEMS [1–4] and VLSI devices [5–8]. Engineers and scientists will need automated techniques that accurately characterize the intrinsic stress in a polymer layer on a wafer being processed to mitigate peeling and device deformation. However, characterizing a polymer layer's intrinsic stress during and after fabrication has proven to be difficult to do in a simple and quick manner. Polymer layer characterization during fabrication is complicated by the requirement that the layer cannot be damaged. One group's effort to nondestructively determine a polymer layer's biaxial tension [9–12] uses a holographic approach to visualize a diaphragms modal pattern stimulated with a single frequency. The problem with the holographic method is that it can take a significant amount of time to collect data and is difficult to fully automate. The purpose of this paper is to present a noninvasive method that solves the difficulties of automating the characterization of the intrinsic tension in a processed polymer layer. Our method requires that a test die be designed into the photomask(s) to form a diaphragm of the polymer layer being investigated. The diaphragm is vibrated using acoustical white noise and the frequency response of the diaphragm is monitored with a laser Doppler vibrometer to determine if the polymer diaphragm acts as a membrane or a plate. If the diaphragm acts as a membrane' the intrinsic stress can be calculated using the resonant peak frequencies obtained with the vibrometer. The reader should note that this method is not appropriate to determine stress in polymer layers that are plate like. The reason this method is not appropriate for plates is that plate behavior is dominated by material characteristics like Poisson's ratio and Young's modulus, but not by biaxial stress. Therefore, the biaxial stress is not easily calculated using this approach for plate like dia-

phragms. The theory, experimental setup, and empirical results obtained from poly dimethyl siloxane (PDMS) are discussed.

Theory

Diaphragms are usually categorized as plates or membranes. Plate like diaphragm motion is linear and is preferable for many sensor applications. Non-linear membrane motion increases the level of sophistication required to determine signals obtained from a membrane like diaphragm.

A diaphragm exhibits plate behavior when the diaphragm's physical properties such as the diaphragm dimensions, density, Young's modulus, and Poisson's ratio dominates the restoring force when the diaphragm is in motion [13–17]. The diaphragms considered in this paper will be circular in nature but the method can be applied to diaphragms of other shapes.

The fundamental frequency of a circular, plate like diaphragm is generally modeled by equation (1) [14].

$$f_{mn} = \frac{\pi \beta_{mn}^2}{2R^2}\sqrt{\frac{Eh^2}{12\rho(1-\nu^2)}} \tag{1}$$

where R is the diaphragm's radius, h is the diaphragms thickness, E is Young's modulus of the diaphragm material, ρ is the density of the diaphragm material, and ν is Poisson's ratio of the diaphragm material. β_{mn} is a Bessel coefficient where m = (0,1,2,3…) and n = (1,2,3,4…). Equation (1) can be further refined to determine the first resonant peak as presented in equation (2) [13].

$$f_{01} = \frac{0.467h}{R^2}\sqrt{\frac{E}{\rho(1-\nu^2)}} \tag{2}$$

The next five resonant frequencies of a diaphragm with plate behavior can be calculated using equation (3) [1].

$$f_{mn} = \mathrm{C}_{mn} f_{01} \tag{3}$$

where f_{01} is the fundamental resonance frequency of the plate and C_{mn} is a coefficient. The first five coefficients of C_{mn} for a plate are C_{11} = 2.09, C_{21} = 3.43, C_{02} = 3.91, C_{12} = 5.98, and C_{03} = 8.75 [1]. The coefficients of the above equations are important because it is these values that the experimental data is compared to. If the experimental data matches the coefficient data of the first few C_{mn} then the diaphragms are plate like and our approach of characterizing the stress in a polymer layer will not work.

A diaphragm exhibits membrane like behavior when tension is the dominant force in the diaphragms' motion [1, 3]. Generally the fundamental frequency of a membrane is modeled with equation (4) [14].

$$f_{mn} = \frac{\beta_{mn}^2}{2R}\left[\frac{S}{m}\right]^{\frac{1}{2}} \tag{4}$$

where S is the diaphragm bi-axial tension, and m is the mass of the diaphragm. Equation (4), like equation (1), can be further refined for determining the first resonant frequency as presented in equation (5) [1].

$$f_{01} = \frac{0.382}{R}\sqrt{\frac{S}{m}} \tag{5}$$

The mass can be further described by equation (6).

$$m = \pi R^2 h \rho \tag{6}$$

The next five resonance frequencies of the membrane like diaphragm can be calculated using equation (3) [1]. The first five coefficients, C_{mn}, for a membrane are C_{11} = 1.59, C_{21} = 2.14, C_{02} = 2.30, C_{12} = 2.92, and C_{03} = 3.60 [1]. Like the plate using equation (3) is important in that the experimental data is compared to the coefficients. If the experimental data matches the coefficients in equations for the membrane case then the diaphragms are membrane like and the biaxial stress of the polymer layer can be determined using our method.

The theoretical outline presented above is an extremely limited overview of diaphragm theory which is presented in more detail in references [13–17]. The reader should be aware that there are special cases in diaphragm theory when the diaphragm has both membrane and plate characteristics [15] and is not addressed in this article.

We determine the fundamental frequencies for the polymer diaphragm under study and divide the f_{mn} frequencies by the f_{01} frequency. The numbers obtained are then compared to the coefficients for the plate and membrane cases. The empirical values are inspected to determine if the sample diaphragm is acting as a plate or diaphragm. If the experimentally obtained numbers agree with the theoretical coefficients for the membrane case then the bi-axial tension is back calculated using equation (5).

Experiment

The samples created to test this method were polymer layers fabricated on a (100) silicon substrate. The

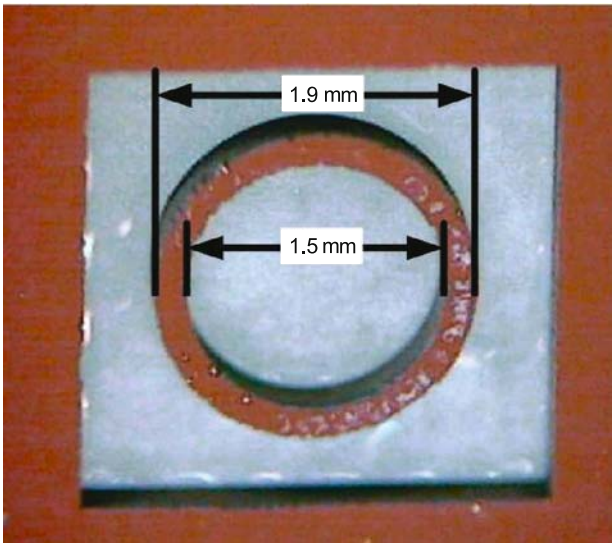


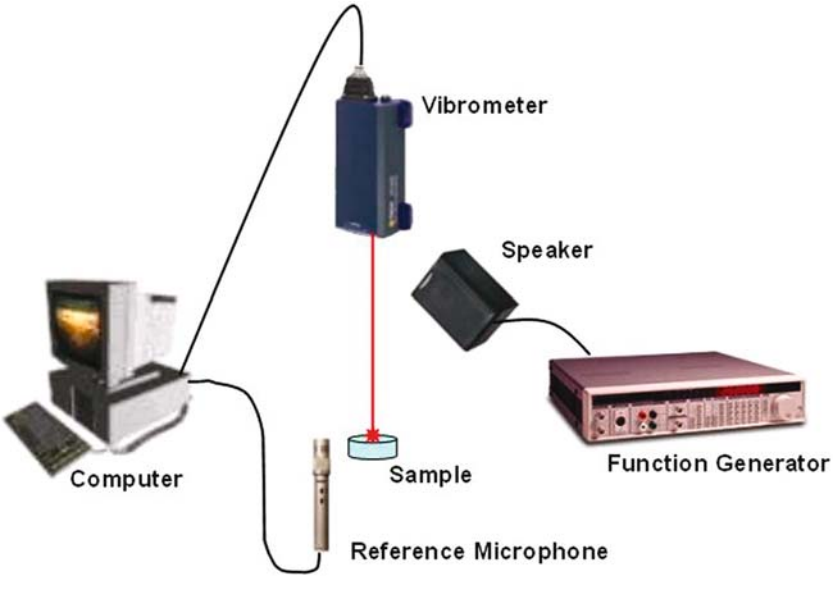
Fig. 1. A testing die, shown from the bottom, used to determine the intrinsic stress in a polymer layer. The dark areas are silicon and the light areas are PDMS. The silicon is 381 mm thick

polymer was released by the use of deep reactive ion etching (DRIE) to remove silicon behind the polymer layer creating the die shown in Fig. 1 with a suspended diaphragm. It is worth mentioning that membranes can be formed in a number of ways and this method will work on a membrane formed in any manner. The polydimethyl siloxane (PDMS) diaphragms were formed by spinning Zipcone™ FN obtained from Gelest Inc (Product number PP1-ZPFN) onto a silicon (100) p type wafer. A spread speed of 500 RPM for 2 sec and a spin speed of 5000 rpm for 30 sec were used to spin the PDMS onto the wafer. The sample was then baked for 45 minutes in a vacuum oven at 100°C in nitrogen ambient. The PDMS diaphragms were 1.5 mm in diameter and had a thickness of 18 um to 20 um determined by using a Tencor contact profilometer. SPR 220 photoresist obtained from Shipley Company was then spun onto the back side of the wafer with a spread speed of 500 rpm for 0.2 sec and a spin speed of 1000 rpm for 20 sec. A soft bake was performed at 100°C for 30 minutes in a vacuum oven. The photoresist was then patterned using contact photolithography, developed in MF 319 obtained from Shipley Company, rinsed in DI water, and dried in nitrogen. The sample then undergoes a STS proprietary DRIE process to etch the silicon from the backside of the wafer to form the diaphragm. The PDMS was used as an etch stop for the DRIE process.

The PDMS diaphragms were then tested using the experimental setup shown in Fig. 2. Before testing of the PDMS diaphragms the system was characterized without a sample to determine vibrations that are due to the test setup. The system frequency response to white noise is presented in Fig. 3.

The speaker, a Foster Personal Monitor 6301B, is centered on and aligned perpendicularly to the samples' surface. A function generator, a Stanford Research Systems Model D5360 Ultra Low Distortion Function Generator, provides the input signal to the speaker. The input signal used was acoustical white noise. The vibrometer used was a Polytec Gmbh Model OFV-353 with the Polytec DFE 650 DSP Front End. The reference microphone, a Shure SM81 Condenser, was connected to the reference microphone port on the DFE 650.

Fig. 2. Experimental setup for acoustically testing membranes



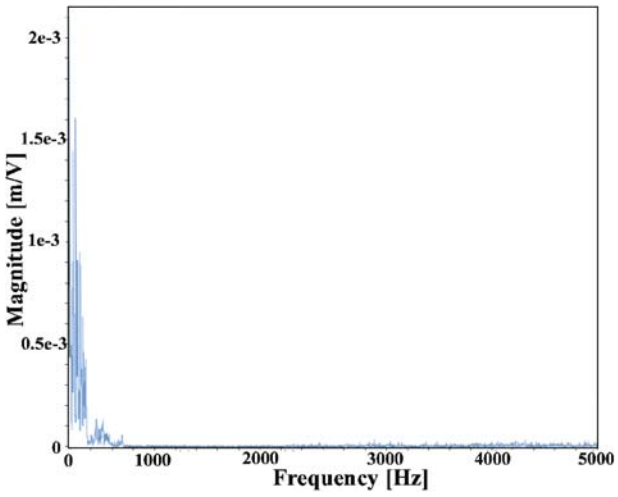


Fig. 3. The frequency response of the test system measured without a sample

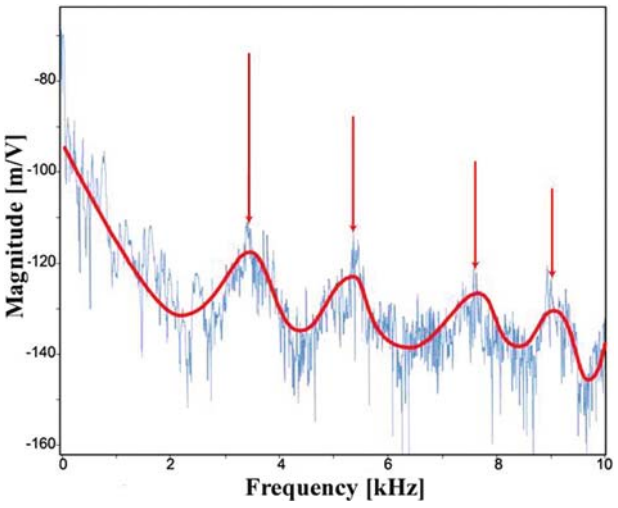


Fig. 4. A plot of a PDMS diaphragm frequency response to acoustic white noise obtained with a laser vibrometer. The line indicates the general curve and arrows show the peaks the authors used in this study

It should be noted that the index of refraction (n) for the PDMS used in this study is 1.45. This is important because the PDMS membrane has a reflectance of approximately 3.5% (similar to the reflection from a glass surface) which is adequate to take measurements with the type of vibrometer used. A reflective layer can be added to a polymer diaphragm to increase reflection but the author cautions that the addition of such a layer can change a MEMS diaphragm significantly. A reflective layer can add new stresses, create corrugations, act as a boss, or mass dampen a small MEMS diaphragm all of which result in significant changes to the diaphragm movement.

There are two sources of error in the current experiment. First, while the laser vibrometer used is perfectly capable of taking accurate readings from low reflectivity surfaces the lower reflectivity of a surface increases the noise in the readings. A surface with a higher reflectivity would improve the signal to noise ratio of the readings. Second, the diaphragm used in this study is attached to a ring of silicon and another irregular shaped diaphragm surrounds the structure. The die design used in this study was designed so that the circular diaphragm could be removed from the silicon wafer for incorporation into another device. Further, an analysis of the weights of the polyimide and silicon structure provides insight into the error, or lack thereof, produced by this geometry. The silicon ring weighs approximately 0.9477 mg and the PDMS weighs approximately 0.06618 mg—more than an order of magnitude difference. One can see that the silicon weight is much larger than the PDMS and because of this it is not probable that the outside structure affects the vibration characteristics of the inside structure. Additionally, it is important to understand that the silicon ring is not "hanging" but is in contact with the test system surface as is the surrounding silicon structure. The authors believe the results would be more accurate if the surrounding diaphragm were removed so that only the circular diaphragm is used.

Results

A typical frequency response for the PDMS diaphragms is shown in Fig. 4.

The peaks of interest are the wide, prominent peaks that are resonant peaks of the diaphragms. The center

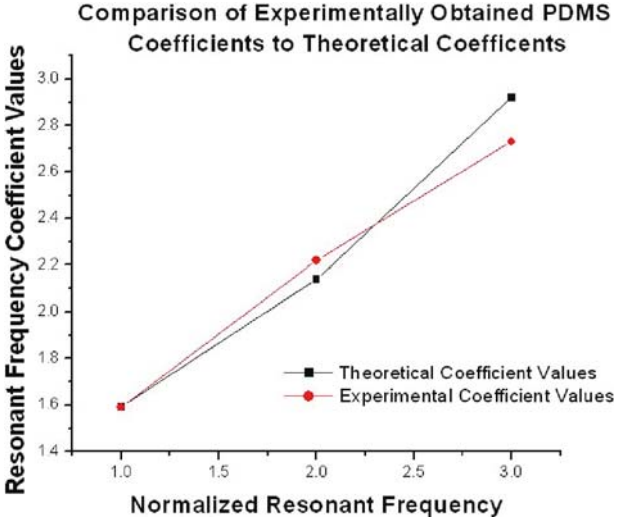


Fig. 5. The average of the experimental results obtained from multiple PDMS diaphragms is compared to the coefficient values reported by Olson

of the resonant peaks was used to calculate the empirical resonant frequency coefficients. The agreement between the experimentally determined resonant frequency coefficients and the theoretical resonant frequency coefficient was found to be strong for the membrane case (see Fig. 5). Solving equation 5 [13] for the tension, S, and substituting in the empirical resonant frequency coefficients allowed the authors to determine the PDMS film intrinsic bi-axial stress to be $1.58 * 10^{-3}$ Dynes/cm. The density of the cured PDMS used was 1.1 g/cc as reported by Gelest. A standard value for the PDMS polymer is not available to compare our empirically obtained value of bi-axial stress to.

Concluding Remarks

A method for determining the intrinsic stress of an on wafer, polymer layer was presented. While the method described in this paper is not destructive or invasive in a manner similar to the approach described in references [9–12], our method has the added advantage that it can be easily automated and tests can be conducted at specific areas on a wafer.

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