High speed wafer scale bulge testing for the determination of thin film mechanical properties

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A wafer scale bulge testing system has been constructed to study the mechanical properties of thin films and microstructures. The custom built test stage was coupled with a pressure regulation system and optical profilometer which gives high accuracy three-dimensional topographic images collected on the time scale of seconds. Membrane deflection measurements can be made on the wafer scale (50–150 mm) with up to nanometer-scale vertical resolution. Gauge pressures up to 689 kPa (100 psi) are controlled using an electronic regulator with and accuracy of approximately 0.344 kPa (0.05 psi). Initial testing was performed on square diaphragms 350, 550, and 1200 μ m in width comprised of 720 ± 10 nm thick low pressure chemical vapor deposited silicon nitride with ~ 20 nm of e-beam evaporated aluminum. These initial experiments were focused on measuring the system limitations and used to determine what range of deflections and pressures can be accurately measured and controlled. Gauge pressures from 0 to ~ 8.3 kPa (1.2 psi) were initially applied to the bottom side of the diaphragms and their deflection was subsequently measured. The overall pressure resolution of the system is good (\sim 350 Pa) but small fluctuations existed at pressures below 5 kPa leading to a larger standard deviation between deflection measurements. Analytical calculations and computed finite element analysis deflections closely matched those empirically measured. Using an analytical solution that relates pressure deflection data for the square diaphragms the Young's modulus was estimated for the films assuming a Poisson's ratio of v=0.25. Calculations to determine Young's modulus for the smaller diaphragms proved difficult because the pressure deflection relationship remained in the linear regime over the tested pressure range. Hence, the calculations result in large error when used to estimate the Young's modulus for the smaller membranes. The deflection measurements of three $1200 \times 1200 \ \mu m^2 \ Si_3N_{4-r}$ membranes were taken at increased pressures (>25 kPa) to increase nonlinearity and better determine Young's modulus. This pressure-deflection data were fit to an analytical solution and Young's modulus estimated to be 257 ± 3 GPa, close to those previously reported in literature. © 2010 American Institute of Physics. [doi:10.1063/1.3427493]

I. INTRODUCTION

The mechanical properties of thin films are an important metric used in design and fabrication of sensors and microelecromechanical systems, and play a large role in overall device behavior.^{1–3} It is well known that thin film material properties vary significantly from their bulk counterparts⁴ and thin films can withstand higher failure stresses than the same bulk material.⁵ Bulge testing is a technique that can quickly derive information on mechanical properties of thin films including: Young's modulus, biaxial modulus, yield strength, fracture strength, and residual stress. In bulge testing, a membrane is fastened into place, pressure is applied, and the resulting deflection is measured. The residual stress can be determined from measuring deflection at low pressures, while the deformations at higher pressures are used to determine the biaxial modulus.⁶ This technique has the unique advantage of extracting these material characteristics simultaneously from membrane deflection.

Data derived from the pressure-deflection relationship are also useful for the development of micropressure sensors. The mechanics that define and stresses induced in the sensing diaphragms are strongly correlated with both deflection and curvature of the bulge. For piezoresistive pressure sensors, it is useful to perform bulge testing to determine sensor sensitivity by correlating their output voltage to applied pressure; the resulting diaphragm deflection can be helpful in determining stress found within the piezoresistors.

In the past, bulge testing thin films was susceptible to sample irregularities (thickness, defects) and a number of problems existed related to sample mounting.⁷ This created a need for samples to be fabricated with tight tolerances and high uniformity. Microfabrication technologies address these requirements directly by using processes that are highly optimized and repeatable. The silicon wafer acts as an excellent substrate for thin films due to its high strength, homogeneity, and wet etching characteristics. Silicon wafers oriented with a [100] surface allow the anisotropic etching along the (110) planes of the substrate using potassium hydroxide (KOH). Many diaphragms can be simultaneously fabricated across the wafer from the deposited thin films which are defined by sidewalls at angles of 54.74° to the surface.

Typically, laser interferometers, atomic force microscopes, and mechanical profilometers have been used to ana-

TABLE I. Initial design specifications of the bulge testing system.

System requirement	Value
Wafer size (mm)	50–150
Diaphragm size (mm)	0.1-2
Measurement time (min)	<1
Deflection range (μm)	0.01 - 1000
Deflection resolution (nm)	<5
Pressure range (kPa)	0-200
Pressure resolution (kPa)	0.5

lyze the deflection of individual films under load.^{8–10} Brown *et al.*,¹¹ hold a patent on a system used to perform bulge testing on the wafer scale using noncontact profilometry similar to the one presented within this report. Alternative methods to calculate stress and strain relationships include x-ray diffraction, nanoindentation, and wafer curvature techniques.^{9,12–15} The American Society for Testing and Materials standard for determination of mechanical properties of bulk materials are tensile and bend tests which prove difficult to perform on thin films.¹⁶

Our bulge test system directly integrates an optical profilometer (Zygo, NewView 5032, Middlefield, CT, USA) and wafer scale mounting stage with a pressure control subsystem (Tescom, ER 3000, Elk River, MN, USA). The apparatus allows the determination of film properties through measuring the deflection of thin diaphragms accurately with a three-dimensional (3D) data set. The initial system specifications are presented in Table I.

This tool also allows us to enhance our thin film deposition techniques and sensor designs by better characterizing thin film mechanical behavior. Large variations in membrane deflection across a wafer indicate that thin film material variation exists either in thickness or microstructure. However, use as an *in situ* monitoring tool requires the backside etching of the wafer after each deposition. This report discusses the design of the apparatus and gives the initial low pressure test results used to determine system capabilities. Analytical and finite element models of membrane deformation are compared empirical measurements. Young's modulus is then calculated for the films using three sizes of square silicon nitride diaphragms.

II. EXPERIMENTAL

A. Bulge testing system

The bulge testing system is comprised of three main components including the diaphragm mounting stage, pressure regulation system, and optical profilometer. The mounting stage is used to the secure wafers ranging in size from 50 to 150 mm and made from aluminum and is presented in Fig. 1.

O-ring grooves were fabricated within the wafer mounting stage with several sizes corresponding to standard wafer diameters including 50, 75, 100, and 150 mm. Silicone o-rings were placed inside the grooves used to seal the backside of the wafers to the stage. This seal was used to maintain pressure on the backside of the wafer while applying pressure. Aluminum clamping rings with flat bottom surfaces



FIG. 1. (Color online) Photograph of the wafer mounting stage used for bulge testing of silicon nitride-aluminum membranes. Wafers ranging in size from 50 to 150 mm can be mounted while pressure is applied to the back-side of the membranes.

approximately 8 mm wide are used to apply uniform pressure to the topside of the wafer circumference creating a compression seal against the o-rings. The aluminum clamping rings are tightened down manually with four screws. This compression insures that small leaks are minimized between the wafer and stage which may lead to pressure fluctuations and deflection error. With this mounting system it is also possible to achieve downward deflection by applying vacuum to the backside of the wafer. Wafer curvature was measured (without applied pressure) using mechanical profilometry (P-20, Tencor, Milpitas, CA, USA) after mounting and showed that the wafer clamping ring compression did not significantly alter the initial wafer curvature. Ports 10 mm in diameter are located in-between differently sized mounting rings are used to apply pressure to the space beneath the wafer. When smaller wafers are tested, ports located on the periphery are sealed using small plugs with integrated o-rings.

Individual test samples can also be mounted to a stainless steel dummy wafer using a two part epoxy. The dummy wafer serves as an "adapter" between the test sample and the wafer level bulge tester. The adapter is made from 304 stainless steel and has an 150 μ m hole located in its center.

Pressure is applied to the mounting stage using an electropneumatic regulator (ER3000, Tescom, McKinney, TX, USA) with the ability to modulate pressures up to 689 kPa (100 psig) with 344 Pa (0.05 psig) accuracy. The regulator was microprocessor controlled and used a proportionalintegral-derivative (PID) method to compare the internal pressure sensor signal to the set point controlling a pair of inlet and outlet solenoid valves. The system has the capability to use a high accuracy external pressure transducer and dome loaded pressure regulator to accurately control pressures over a wider range. Internal control parameters such as PID settings, zero, and span can be remotely controlled via a personal computer through a RS-485 interface. Data recording and custom pressure routines can also be easily implemented. A Newview 5032 (Zygo Instruments Inc., Middlefield, CT, USA) system was used to perform noncontact white light optical profilometery on deflected membranes. The system has the advantage of making fast measurements (<10 s) without physical contact and able to make lateral measurements over the 150 mm diameter wafer. Our instrument had the 5× and 20× objectives installed which allows us to analyze diaphragms 250 μ m to 2.5 mm in size and deflections in the nanometer range. This report only discusses square diaphragms that are from 350 to 1200 μ m in width bulged at low pressure (<10 kPa)

B. Membrane preparation and characterization

The Si_3N_{4-x} films used in this study were deposited on *n*-type $(1-10 \ \Omega \text{ cm})$ 100 mm diameter silicon wafers by low pressure chemical vapor deposition (LPCVD). This LPCVD nitride also acts as the KOH bulk silicon etch mask. The deposition was performed at 825 °C for 2 h with an approximate deposition rate of 6 nm/min. The precursors used were dichlorosilane (DCS) and ammonia at a 6:1 ratio (DCS:NH₃) with flow rates of 60 and 10 SCCM (SCCM denotes cubic centimeter per minute at STP), respectively. The stoichiometry of the Si:N is 1.0:1.07 according to x-ray photo electron spectroscopy data. An average Si_3N_{4-x} tensile stress was estimated using Stoney's equation¹⁷ and found to be 311 ± 7 MPa for the initial wafers tested at low pressures. A second LPCVD deposition was performed using the same deposition parameters for higher pressure deflection testing to estimate the Young's modulus, these wafers had an average residual stress of 165 ± 5 MPa. Stress in these films is attributed to the mismatch between the coefficients of thermal expansion of the Si substrate and the film and intrinsic stress within the film.¹⁸ Wafers with deposited LPCVD nitride were coated with Shipley 1813 positive photoresist was spun on at 3000 rpm for 30 s and soft baked for 90 s at 110 °C. Three sizes of square etch openings were patterned on the wafers using chrome masks which corresponded to diameter of 350, 550, and 1200 μ m. Alignment of the square diaphragms to the wafer flat was performed using an Electronic Visions 420 (EV Group Inc., Tempe, AZ, USA) mask aligner on the backside of the wafer. Each of the wafers was patterned with an orthogonal array membranes aligned to the wafers edge with a pitch of 5 mm. The silicon nitride provides high selectivity when used as a potassium hydroxide (KOH) masking layer and acts as the structural material for free-standing square membranes in this experiment. The photoresist was exposed using an UV light source for 9 s with an intensity of 75 mJ/cm² for patterning. Then it was developed using Shipley 352 developer for 1 min. Resist was hard baked for 5 min at 90 °C. Reactive ion etching was performed using an Oxford 100 at a pressure of 5 mTorr and power of and 100 W. A mixture of SF₆ and O₂ are used to open etch windows on the backside of the wafer.

A Tencore P-20 (Tencor, Milpitas, CA, USA) contact profilometer verified a nitride film thickness of 720 ± 10 nm measuring across the windows. Initial wafer thickness measurements using a dial indicator (series-543, Mitutoyo, Kanagawa, Japan) yield a total wafer thickness of $423 \pm 1 \mu$ m. A square opening was wet etched through the backside of the wafer using 60% concentration KOH solution at 87 °C. The backside openings were etched for 7.75 h giving an average etch rate of ~1.1 μ m/min. The Si₃N_{4-x} membranes appeared yellow and semitransparent. Due to the transparency the Si₃N_{4-x} membranes optical profilometery could not be used directly on the membranes, therefore e-beam evaporation of 20 nm of aluminum was performed in a Denton evaporator (Denton Vacuum, Moorestown, NJ, USA) system at a voltage of 6.5 kV and current of 0.1 A to make the membranes opaque. An approximately 20 nm thick film was deposited in 15 s at a rate of at a rate of ~13 A/s, measured using a quartz crystal thickness monitor.

III. TESTING METHODS

Wafers with silicon nitride diaphragms were loaded into the bulge testing stage and mounted in the Zygo Newview 5032 motorized XY table. The membranes were aligned under the microscope objective in field of view and then placed into focus. Pressures were applied on the backside of the membranes and all scans were performed using the 20× objective and zoom of $1.3\times$ with a scan height of 20 μ m. The membrane height data were recorded at pressure intervals of 645 Pa (0.1 psi). After measurements were complete, data were exported using Zygo METROPROTM software which allows a two-dimensional cross section to be taken in the diaphragm center.

IV. RESULTS AND DISCUSSION

A. Analytical model

A number of analytical models have been developed to study deflection and stress characteristics in square membranes as a function of applied pressure. The relationship between the external pressure and the membrane deflection initially was studied by Timoshenko who developed an analytical solution. There also exists an nonlinear solution of the pressure-deflection relationship presented by Levy in the form of a series, which is also given in Timoshenko's book on plates and shells.¹⁹ Given the series form of the solution and the difficulty in determining integration constants it is not convenient for the analysis of bulge test results. Tabata²⁰ was able to calculate biaxial modulus and Poisson's ratio from deflection characteristics of rectangular membranes. Pan *et al.*²¹ compared the analytical solution with finite element method analysis and found that the functional form of the analytical solution is correct, but the constants needed minor correction. Several additional models were also developed to improve accuracy of material characterization from the load-displacement data.^{6.7,10,22-24} Maier-Schneider et al.²⁵ developed a solution in determining pressure-deflection characteristics for square silicon nitride membranes shown in Eq. (1). This solution is similar in form to more current solutions found by Vlassak and co-workers^{7,26}

$$P = 3.45 \left(\frac{\theta \cdot t}{a^2}\right) d + 2.48 \left(\frac{Et}{a^4}\right) d^3,\tag{1}$$

where *P* is the load pressure, *t* the thickness, *E* Young's modulus, σ_r the residual stress, 3.45 and 2.48 were empirically found numerical constants that depend on Poisson's

TABLE II. Material parameters used in COMSOL 3.3 finite element simulations.

Simulation parameter	Value
Modulus Si ₃ N _{4-s} (GPa)	297
Si ₃ N _{4-e} residual stress (MPa)	311 ± 7
Modulus Al (GPa)	79
Membrane width (μm)	350-1200
Si_3N_{4-x} thickness (nm)	720
Al thickness (nm)	20

ratio and membrane aspect ratio, and *d* is the maximum center deflection at one half of the membrane's edge length. The aluminum layer is less than 5% of the membrane thickness and Young's modulus is significantly lower (70 GPa),¹⁶ therefore this model is still applicable although it neglects the additional reflective aluminum layer.

B. Computer simulations (finite element analysis)

Finite element analysis (FEA) was carried out on the Si_3N_{4-x} /Al membrane structures using COMSOL 3.4A (Comsol, Burlington, MA, USA). One quarter of the square membrane is modeled because fourfold symmetry can be utilized to reduce the complexity of the model and reduce total number of calculations. Since the membranes are subjected to relatively low pressures the deformation of silicon wafer support frame can be neglected as determined by initial simulations. Fixed boundary conditions are placed on two of the adjacent membrane edges (clamped) mimicking the silicon wafer frame. On the remaining two internal edges symmetry boundary conditions were used. For analysis, the membrane was first meshed using square mapping then divided into rectangular elements in all three dimensions. This meshing was used because it is the most efficient in terms of node quantity and calculation efficiency for this geometry. Large deformation conditions were utilized since the deflections of the membranes were several times that of the membrane thickness over the simulated pressure range. According large defection theory, the work created by the application uniform pressure on the membrane is transformed to the elastic energy of the membrane, which consists of the material stretching.²⁷ The stretching is due to the extending of the middle plane of the membrane and bending due to the outof-plane displacement. Material parameters used for computer simulations are shown in Table II.

The static pressure load is applied to backside of the membrane up to 8.28 kPa (1.2 psi) in increments of 0.689 kPa (0.1 psi). A parametric study was used to determine the deflection over this pressure range the deflection results at \sim 6.2 kPa (0.9 psi) are shown in Fig. 2.

C. Empirical measurements

Accurate deflection measurements of thin films require that low pressures (<10 kPa) can be applied without the interference of noise from the outside environment. The primary objective of these initial tests was to qualify the system and determine any sources of error and/or noise. Deflections data can be displayed in a number of different formats in-



FIG. 2. (Color online) Displacement plot illustrating a deflection of 2.68 μ m for a quarter of the $1200 \times 1200 \ \mu$ m² square membrane calculated using FEA at a pressure of 6894 Pa (1 psi). The composite structure consists of 720 nm of silicon nitride with 20 nm of aluminum. The bottom and right edges are fixed (clamped) and a symmetry boundary condition placed on the left and top sides.

cluding 3D mesh plots, solid plots, and surface plots shown in Fig. 3. The optical image of the bulged 1200 \times 1200 μ m² diaphragm shows that aluminum layers were deposited with large compressive stress, consistent with the delamination and buckling observed in the Fig. 3(b).

Due to the significant aluminum film buckling and the thickness of the layer being approximately 2.7% of silicon nitride we disregarded the 20 nm sputtered aluminum layer for analytical calculations. The finite element simulations took the aluminum layer into account but assumed the aluminum residual stress was negligible. The deflections for the



FIG. 3. (Color online) Height data displayed as (a) 3D plot. (b) solid plot. and (c) surface plot from the Zygo Newview 5032 optical pyrometer. This membrane is 1.2 mm length and a laminate structure consisting of 720 nm of Si_3N_{4-r} and 20 nm of aluminum loaded at 6894 Pa (1 psi).



FIG. 4. (Color online) Comparison of the theoretical. simulated, and measured defection of diaphragms with widths of (a) 350 μ m, (b) 550 μ m, and (c) 1200 μ m at applied pressures ranging from 689 Pa (0.1 psi) to 6894 Pa (1.2 psi). Figure 4(d) shows that standard deviation between measurements is a function of pressure and diaphragm sensitivity.

square diaphragms 350, 550, and 1200 μ m in width are presented in Figs. 4(a)-4(c) for pressures ranging from 0 to 8.3 kPa (1.2 psi). Deflection results show little variation between theoretical, simulated, and measured results. Figure 4(d) shows the standard deviation between measurements for the various membranes.

When comparing the analytical and FEA diaphragm defection models, FEA deflections were slightly lower, which may be due to the additional aluminum layer that had to be added on top of the diaphragm. In general, the deflections of the membranes were slightly lower than analytical calculations but higher than simulations and within 2% of calculated values. We additionally observed through data analysis that minute pressure fluctuations impacted the deflection data and a number of trends existed. First, as the membranes become larger they become more susceptible to minute pressure fluctuations and the membranes have a higher standard deviation between identical measurements shown in Fig. 4(d). Second, at lower pressures, measurements have higher variation in deflection. We suspect this discrepancy in measurements is caused by pressure variations due to control limitations of the electronic pressure regulator. Since the deviation of empirical measurements is small (10^{-2}) with respect to the mean deflection values this system is capable of measuring minute deflections at low pressures (<10 kPa) reliably.

In order to study film variability across the wafer, three diaphragms of each size were on the edges and center of the wafer were characterized using a pressure of 6.89 kPa (1 psi). Results from this analysis are presented in Fig. 5(a) showing the system is capable of making measurements on

the wafer scale with high resolution. No trends appear to exist in relation to wafer position and the deflection values have similar standard deviations when compared to previous samples.

The system allows the export of data from the cross section of the bulged diaphragm in order to analyze the shape of the deflected diaphragms. Although these data can potentially become slightly skewed with respect to the origin of the coordinate system since the user manually defines the centerline of the bulge, deflection results closely match those predicted by FEA. Any slight misalignment of exported bulge shape could possibly be alleviated with software routines that automatically determine the membrane centerline. The shape of the deflected diaphragms generated by the FEA models was also compared with the real membrane shape along the centerline shown in Fig. 5(b). The comparison of these curves proves useful in predicting different deformation behaviors. This comparison can help validate experimental uncertainties and establish which models fit the deflection shape most accurately. This substantiates boundary conditions used within the model and gives confidence when other analyzing parameters such as stress and strain.

V. ESTIMATION OF YOUNG'S MODULUS

Since testing was performed on square membranes the estimation of Poisson's ratio is required in order to solve for the Young's modulus using Eq. (1). A value of ν =0.25 was used for Poisson's ratio which leads to values of 3.45 and 2.48 for the constants of Eq. (1). We used measured values of



FIG. 5. (a) Deflection measurements taken for samples located on the left, right, and center wafer at a pressure of 6.2 kPa (0.9 psi). (b) Comparison of the measured (---), simulated (- - -), and analytical (\bullet) deflection of a 1200×1200 μ m² square silicon nitride-aluminum membrane with load ranging from 689 to 6.2 kPa (0.1 to 0.9 psig). Analytical and simulated results show little variation.

residual stress ($\sigma_r = 311 \pm 7$ MPa) and thickness (t = 720 nm) for these calculations, which should be similar between each of the samples found on the test wafer. Therefore we can use Eq. (1) to estimate the Young's modulus for deflections of the various membrane sizes as shown in Fig. 6.

We determined at lower pressures (<10 kPa) it was difficult to estimate the Young's modulus for all diaphragms. Smaller diaphragms at these low pressures were only found to deflect mainly in the linear regime; therefore calculation of the Young's modulus was inaccurate with high error. Specifically the $1200 \times 1200 \ \mu m^2$ diaphragm had average calculated Young's modulus ranging from 321 to 576 GPa from 4.5 to 8.5 kPa, well above values found in literature. While trying to calculate the Young's modulus for low pressure data we determined that a combination of inability to control low pressures exactly and limitations in the submicron resolution of the optical profilometer created incorrect or shifted modulus results for many of the individual data points.

In order to more accurately predict the Young's modulus additional experiments were performed on three LPCVD Si_3N_{4-y} diaphragms ($1200 \times 1200 \ \mu m^2$) at increased pres-



FIG. 6. (Color online) Calculation of Young's modulus using Eq. (1) for diaphragms with widths of 350, 550, and 1200 μ m over a pressure range of 0 to 8.5 kPa (0 to 1.23 psi). Calculated values for Young's modulus at these low pressures were nonsensical due to the high measurement variability attributed to reduced pressure control and limitations of instrument resolution. The largely linear deflections of the diaphragms do not allow for accurate calculation of the films Young's modulus.

sures (0 to 26.2 kPa) on samples deposited during the second LPCVD deposition. These films had approximately the same thickness (715 ± 3 nm), confirmed using ellipsometry and average residual stress of 165 MPa. The deflection pressure relationship was plotted and fit using Eq. (1) across the entire range of measurements as shown in Fig. 7. The fit was accurate with r-square values >99% and this technique showed improved outcomes over low pressure results methods described above. The average Young's modulus for the three samples was fit across the entire pressure range and was calculated to be 257 ± 3 GPa for the $1200 \times 1200 \ \mu m^2$ diaphragms. This estimate for Young's modulus appears in good relation to values reported in literature. Fitting of the pressure-deflection function [Eq. (1)] achieved the best results with the empirically measured data for the largest diaphragms at highest pressures. It was determined that the system was incapable of estimating the Young's modulus of



FIG. 7. (Color online) Fit of analytical solution [Eq. (1)] to the pressure deflection data of three Si₃N_{4-x} diaphragms ($1200 \times 1200 \ \mu m^2$) at elevated pressures (0–26.2 kPa). The pressure deflection relationship is more nonlinear and the fit estimates Young's modulus (e) of the films to be 257 ± 3 GPa.

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smaller diaphragms, due in part to primary being in the linear deflection regime. Therefore future tests should implement higher pressures and larger diaphragms to increase deflection nonlinearity. This nonlinearity will increase the contribution of Young's modulus on the membrane deformation, which will allow more accurate quantification of this value.

VI. CONCLUSIONS

We design, fabricated, and tested a bulge testing system that can rapidly measure deflections and curvature of thin membranes principally on Si at the wafer scale over a large range of pressures. The performance of this system was characterized by measuring the bulge deflection of thin silicon nitride diaphragms 720 nm thick with a reflective e-beam evaporated aluminum layer (20 nm) at low pressures (<8.3 kPa). Deflection results from the silicon nitride membranes with a reflective sputtered aluminum layer show excellent correlation between the empirical membrane deflection measurements and analytical and FEA model results. The overall pressure resolution of the system is good $(\sim 350 \text{ Pa})$ and small fluctuations exist at pressures below 5 kPa leading to a larger standard deviation between deflection measurements at low pressures. Deflection measurements taken at various locations across the wafer showed have little variation and similar standard deviations between measurements. Unfortunately for the smaller diaphragms measured deflections were in the linear regime and therefore an accurate calculation of Young's modulus was unsuccessful. Higher pressure tests were performed on three 1200 $\times 1200 \ \mu m^2$ diaphragms with lower measured residual stress (165 MPa), experimental fit of the data to an analytical solution gives r-square value >99% and Young's modulus of 257 ± 3 GPa.

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