Optomechanical characterisation of compressively prestressed silicon oxynitride films deposited by plasma-enhanced chemical vapour deposition on silicon membranes

J. Jozwik, P. Delobelle, C. Gorecki, A. Sabac, L. Nieradko, C. Meunier, F. Munnik

To cite this version:

J. Jozwik, P. Delobelle, C. Gorecki, A. Sabac, L. Nieradko, et al.. Optomechanical characterisation of compressively prestressed silicon oxynitride films deposited by plasma-enhanced chemical vapour deposition on silicon membranes. Thin Solid Films, Elsevier, 2004, 468 (1-2), pp.84-92. 10.1016/j.tsf.2004.04.019 . hal-00096940
Optomechanical characterisation of compressively prestressed silicon oxynitride films deposited by plasma-enhanced chemical vapour deposition on silicon membranes

M. Józwik\textsuperscript{a,b}, P. Delobelle\textsuperscript{c}, C. Gorecki\textsuperscript{a,*}, A. Sabac\textsuperscript{a}, L. Nieradko\textsuperscript{a}, C. Meunier\textsuperscript{d}, F. Munnik\textsuperscript{e}

\textsuperscript{a}Laboratoire d’Optique P.M. Duffieux (UMR CNRS 6603), Université de Franche-Comté, 16 Route de Gray, 25030 Besançon Cedex, France
\textsuperscript{b}Institute of Micromechanics and Photonics, Warsaw University of Technology, Św. A. Boboli 8 St., 02-525 Warsaw, Poland
\textsuperscript{c}Laboratoire de Mecanique Appliquée R. Chaleat (UMR CNRS 6604), Université de Franche-Comté, 24 rue de l’Epitaphe, 25000 Besançon Cedex, France
\textsuperscript{d}Centre de Recherches sur les Ecoulements Surfaces et Transferts (UMR CNRS 6000), Université de Franche-Comté, 4 place Tharradin, 25200 Montbéliard Cedex, France
\textsuperscript{e}Centre d’Analyses par Faisceau Ionique, 8A rue Jambe Ducommun, 2400 Le Locle, Switzerland

Abstract

We have studied the composition and physical properties of silicon oxynitride (SiO\textsubscript{x}N\textsubscript{y}) films grown on silicon membranes by means of plasma-enhanced chemical vapour deposition (PECVD process). The influence of these properties on the mechanical behaviour of PECVD deposited films is investigated by an original “point-wise” deflection method. This technique, particularly appropriate for determining the residual stress in the case of prestressed membranes, is operating at the first order of buckling. It combines the classical interferometry with the nanoindentation technique. The interferometry measures the out-of-plane displacements of membranes with SiO\textsubscript{x}N\textsubscript{y} layers of different optical quality, while the nanoindentation permits the extraction of micromechanical properties such as hardness and Young’s modulus. From the “point-wise” deflection technique, the distribution of residual stress was monitored as a function of the refractive index of SiO\textsubscript{x}N\textsubscript{y} films, establishing the correlation between the optical and micromechanical properties of deposited thin films. High measuring accuracy and resolution have been demonstrated, allowing the measurements to be used to enhance PECVD process control.

Keywords: PECVD process; Silicon oxynitride; Buckled membrane; Optical properties; Stress evaluation

1. Introduction

The tendency towards continuous miniaturisation of microelectromechanical systems (MEMS) is a further challenge to the thin films in use [1]. Optical waveguides may be integrated with MEMS structures such as beams, bridges and membranes. In such micro-optoelectromechanical systems (MOEMS), the waveguide can play the role of a sensor to detect a physical parameter of mechanical deformation. Such optomechanical structure is fabricated using bulk micromachining, where a number of different thin films is deposited on top of the silicon wafer, and the desired microstructures are obtained by successive deposition, etching and patterning operations on the surface of the wafer. The fabrication process induces additional internal and thermal stresses between different materials and strongly influences the mechanical behaviour and long-term performance of such microparts. For these reasons, easy, nondestructive and high-resolution measurement of micromechanical properties is crucial for optimising the design and defining the process control of MOEMS devices. Various mechanical techniques have been proposed to evaluate the simple properties such as Young’s modulus, residual stress, strain and Poisson’s ratio in the material [2–4]. Implementing of these methods is difficult in the case of thin film materials, and the reported values of micromechanical parameters vary widely, demonstrating that the

* Corresponding author. Tel.: +33-03-816-66607; fax: +33-03-816-6423.
E-mail address: christophe.gorecki@univ-fcomte.fr (C. Gorecki).
measurement accuracy is not enough to provide high-confidence testing tools. Thus, the reported values of Young’s modulus for polysilicon have ranged from 120 to 201 GPa. The potential of nondestructive optical techniques was also demonstrated in the determination of local material properties and dynamic investigation of MEMS/MOEMS. The efficiency of conventional or phase-shifting interferometry [5], holographic interferometry and digital holography [6], white light stroboscopic interferometry [7], speckle interferometry [8] and electronic speckle pattern interferometry [9] has been demonstrated.

Plasma-enhanced chemical vapour deposition (PECVD)-grown SiO$_2$, films can be applied as a core layer for channel waveguides [10,11]. However, the use of PECVD is still limited here because the residual stress of deposited films is difficult to control, and there is a limited understanding of film properties. In particular, the evaluation of mechanical parameters from compressively prestressed membranes suffers from absence of validated mechanical models. In order to improve this situation, we have firstly studied the material structure and composition of PECVD silicon oxynitride films by Rutherford backscattering spectroscopy (RBS), elastic recoil detection analysis (ERDA) and X-ray reflectivity (XRR). Secondly, we reported the influence of these properties on the mechanical behaviour of PECVD SiO$_2$ films. For this purpose, the evaluation of mechanical parameters is performed in case of buckling results with those reported in literature.

In order to determine electronic densities of SiO$_2$ films, an He$^+$ beam was generated with an angle between sample normal and incoming beam of 76° and the analysis spot size is $0.5 \times 0.5$ mm$^2$. The scattered ions were detected with charge-sensitive “surface barrier” detectors at an angle of 150° for RBS and 20° for ERDA. An additional RBS measurement with a sample angle of $-30°$ has also been performed, and for sample a, RBS measurement with 2 MeV protons has been done. RBS with two different sample angles was used to obtain information on the composition and thickness. ERDA was used to obtain the depth profile for hydrogen. We used an X-ray system to determine electronic densities of SiO$_2$ films, using a Philips X’Pert MPD equipment. Experimental data are summarised in Table 2 where the film thickness, the atomic percent of silicon, nitrogen, oxygen and hydrogen, and mass density of deposited films with various refractive indexes are given.

For samples (a, b, d, e, f) of SiO$_2$, the etch rate is measured in a mixture for isotropic etching of HF (50%):HNO$_3$ (69%):H$_2$O with a volume ratio of 9:75:50, operating at room temperature.

### 2.3. Methodology of SiO$_2$ membrane fabrication

Five sets of square (3.6 $\times$ 3.6 mm$^2$) SiO$_2$ membranes were fabricated, as shown in Fig. 1 [13]. Firstly, both sides of a double-polished (100) silicon wafers was thermally oxidized at the temperature of 1050 °C to grow 1-μm-thick layer of oxide. Square mask aligned in (110) direction was patterned by photolithography on the topside of the wafer. Then, the oxide layer was removed in buffered HF etch. The silicon substrate from which the oxide layer has been

---

**Table 1**

| Sample | $R$=($N_2O$)/($N_2O$+NH$_3$) | N$_2$O [sccm] | SiH$_4$ [sccm] | NH$_3$ [sccm] |
|--------|-------------------------------|---------------|---------------|---------------|
| a      | 1                             | 143           | 7             | 0             |
| b      | 0.88                          | 126           | 7             | 17            |
| c      | 0.60                          | 86            | 7             | 57            |
| d      | 0.32                          | 46            | 7             | 97            |
| e      | 0.11                          | 17            | 7             | 126           |
| f      | 0                             | 0             | 7             | 143           |
removed was anisotropically etched by a KOH solution, giving a 15-μm-thick membrane. After photoresist stripping on the topside of the wafer, the oxide layers were removed from both sides. Finally, 2-μm-thick SiO<sub>x</sub>N<sub>y</sub> films were deposited by PECVD on the backside of the wafer, operating under PECVD conditions described in Section 2.1. In the following sections, letters B, C, D, E, F denote the range of refractive index for each set of SiO<sub>x</sub>N<sub>y</sub> prestressed membranes.

2.4. Evaluation of mechanical properties of SiO<sub>x</sub>N<sub>y</sub> thin films

In this section, Young’s modulus and residual stress state of SiO<sub>x</sub>N<sub>y</sub> of membranes with different optical quality are characterised with the following investigations: (a) estimation of micromechanical properties of membranes (Young’s modulus, hardness) by nanoindentation tests; (b) evaluation of residual stress by means of membrane “point-wise” deflection method using the interferometry; and (c) analysis by means of finite element method (FEM).

2.4.1. Methodology of nanoindentation test

To measure the Young’s modulus and hardness of PECVD-grown SiO<sub>x</sub>N<sub>y</sub> membranes, we applied the indentation tip of Nano-Indenter IIs on the bulk part of a sample containing 2-μm thick SiO<sub>x</sub>N<sub>y</sub> film on top of 380-μm-thick silicon substrate. The tip is applied during the well-controlled loading–unloading cycles. The applied load \( P \) of the indenter is measured as a function of its penetration depth \( h \), delivering the “load–displacement curve” from which Young’s modulus \( E \) and the hardness \( H \) of sample are calculated [14,15]. In addition, we used the “continuous contact stiffness method”, where the penetration of the indenter tip into the sample is accompanied by 1 nm amplitude vibration of the tip performed at the frequency of 45 Hz [15]. This permits to follow the variations of Young’s modulus \( E \) and hardness \( H \) as a function of indentation depth. The maximum depth of indentation is fixed at 900 nm and 15 indents are performed for each of five samples on the test area of 300 × 300 μm<sup>2</sup>.

2.4.2. Methodology of deflection measurement by interferometry

The compressive buckling due to PECVD deposition produces an initial deflection \( h_0 \). To measure \( h_0 \) at the center of SiO<sub>x</sub>N<sub>y</sub> membranes, we used an interferometric technique, shown in Fig. 2 [16]. This is based on a metallurgical Nikon microscope where a Twyman–Green interferometer (TGI) is inserted between the sample to be measured and the microscope objective. TGI consists of a cube beamsplitter where one of beamsplitter facets is metal overcoated, playing the role of the reference mirror of the interferometer. The sample is illuminated by a He–Ne laser beam \( (\lambda = 632.8 \text{ nm}) \) obtained by a spatial filter and directed through a collimating lens to the cube beamsplitter. Both the reference and measuring arms produce interference patterns. Five interferograms with \( \pi/2 \) relative phase shifts between the measuring and reference wavefronts are acquired and visualised by a 768 × 576 CCD array coupled with a frame grabber and then was analysed by automatic fringe pattern analyser [17]. Once continuous phase distribution \( \Phi(x,y) \) is evaluated, the corresponding out-of-plane displacement \( h_0(x,y) \) is calculated with inaccuracy within 10 nm.

3. Theory behind the FEM analysis of the optomechanical evaluation of residual stress

The principle of stress evaluation in thin films is based on the measuring the point-wise deflection at the membrane center. The nanoindenter tip is applied on the membrane
with increased load \( P \), and deflection \( h \) produced by the tip is monitored. This permits the calculation membrane stiffness \( S = dP/dh \). Therefore, knowing the Young’s modulus and Poisson ratio, as well as the analytical relationship between the load \( P \) to the membrane deflection \( h \), the presence of residual stress, the average value of stress is extracted. The efficiency of “point-wise” deflection techniques, less widely used than the bulging test [18–21], was demonstrated in case of tensile membranes [12,22], where the membrane is not fixated by fluid-tight or airtight gluing on a rigid support, modifying the state of residual stress. However, the interpretation of experimental results, obtained by deflection method on compressively prestressed materials, such as of SiO\(_2\), films, is difficult because the absence of theoretically validated models. Consequently, we report here the experimental results accompanied by the theoretical study as well as the modeling data obtained by finite element method (FEM).

Interferometric data demonstrated that the square SiO\(_2\) membranes are operating at the first mode of buckling (Fig. 3). In this case, the theoretical deformation at first approximation is [23]:

\[
h = \frac{-h_0}{4} \left( 1 - \cos \frac{2\pi x}{a} \right) \left( 1 - \cos \frac{2\pi y}{a} \right)
\]

where \( a \) is the size of the membrane. A more accurate second-order relation was reported by Maier-Sneider et al. [24].

**3.1. Theoretical study of the relation \( P=f(h) \) in case of bimorph membranes**

In the case of bimorph membranes, an isotropic material, such as a SiO\(_2\) substrate of silicon, is grown on top of anisotropic (100) substrate of silicon, generating an internal stress of compressive or tensile nature. When the magnitude of stress attain the value of critical stress of buckling, the bimorph structure presents an initial curvature. If the exact analytical solution of this problem is impossible for square membranes, the analytical solutions can be calculated by FEM in case of circular geometry. This last solution can be then extended to the case of square geometry. It was reported [23] that the point deflection at the center of a square membrane containing one material is:

\[
P = \frac{D}{8a^2} h + C \left( \frac{v - e}{e} \right) \frac{Ee}{a^2} h^3 \text{ with } D = \frac{Ee^3}{12(1 - v^2)} \quad (2)
\]

where \( D \) is the bending moment, \( E \) and \( v \) are Young’s modulus and Poisson’s ratio, \( e \) is the membrane thickness, \( a = 5.6 \times 10^{-3} \) is a parameter for square membranes, and \( C \) is a function of \( v \) and \( h/e \) calculated by FEM. For small values of \( h/e \), we have \( C = 6.1/(1 - v^2) \) [23]. For small deflections with \( h/e < 0.3 \), the function \( P=f(h) \) is linear. When the deflection is several times higher than the membrane thickness, \( P=f(h) \) follows a cubic law.

In the case of bimorph membranes, the effective parameters \( D_{eff}, E_{eff}, \) and \( e_{eff} \) are defined. For anisotropic materials, \( D_{eff} \) becomes a function of the bending moments \( D_{eff, i} \) and \( H_{eff,i} \) these bending moments depending from the elastic constants \( C_{ij} \) and crystallographic orientations of material. For the membranes of circular geometry with uniform distribution of stress \( \sigma_{0} \), a “stress function” \( g(k_0) \) is defined. This function is defined for circular membranes of radius \( a \) in both tensile or compressive situations. Then, the solutions will be recalculated by FEM for square geometry of membrane with:

\[
k_0^2 = \frac{N}{D_{eff}} \quad \text{with} \quad N = \sigma_t e_t + \sigma_s e_s = \sigma_{eff} (e_t + e_s) \quad (3)
\]

where \( N \) is the radial force applied by unit of length and linking the substrate stress \( \sigma_s \) with film stress \( \sigma_t \) and \( \sigma_{eff} \) represents the effective stress.

In the case of tensile stress [12,22], we have:

\[
g(k_0) = \frac{8}{k_0^2} \left[ \frac{K_1(k_0) - \frac{1}{k_0}}{I_1(k_0)} (I_0(k_0) + 1) \right. \\
+ \left. K_0(k_0) + \ln \frac{k_0}{2} + \gamma \right] . \quad (4)
\]

In the case of compressive stress, we obtain:

\[
g(k_0) = \frac{8}{k_0^2} \left[ \frac{\frac{\pi}{2} Y_1(k_0) + \frac{1}{k_0}}{J_1(k_0)} (1 - J_0(k_0)) \right. \\
+ \left. \frac{\pi}{2} Y_0(k_0) - \ln \frac{k_0}{2} - \gamma \right] . \quad (5)
\]

\( K_0/K_1 \) are Bessel functions of the second kind of orders \( 0/1 \), \( I_0/I_1 \) are the modified Bessel functions of the first kind of orders \( 0/1 \), \( \gamma \) represents the Euler’s constant, \( Y_0/Y_1 \) are...
the Bessel functions of the first kind of orders 0/1 and \( J_0/J_1 \) are the Bessel functions of the second kind of orders 0/1.

In compressive situation, the first zero of the function \( J_1(k_0) \) is equal to the value \( k_0^{cr} \) of the critical stress of buckling. The \( g(k_0) \) is plotted in Fig. 4, where the different branches represents three situations of stress: (a) tensile stress with \( \sigma_{eff}<0 \) and \( 0<g(k_0)<1 \); (b) compressive stress before the buckling with \( \sigma_{eff}>0 \) and \( 1<g(k_0)<\infty \); and (c) buckling regime of membrane for \( \sigma_{eff}<0 \) and with \( -\infty<g(k_0)<0 \). A FEM modeling of these functions was performed on circular and square membranes, as shown in Fig. 4, where the calculation results are reported with circular or square symbols. Excepted for high values of \( k_0 \) around \( g(k_0)=0 \), there is a good agreement with analytical data. Around \( g(k_0)=0 \), FEM model is not working.

In conclusion, the analytical solution of \( g(k_0) \) is validated for small deflections, while for the strongest values of \( k_0 \), the analytical solution is not applicable because the buckling produces an initial deflection of membranes that can be above the membrane thickness. The transposition of these results in the case of square membranes requires the introduction of corrective factor on the parameter \( k_0 \). This is calculated by FEM modeling and validated in both compressive and tensile [19] situations (see Fig. 4):

\[
g(k_0) = \frac{2k_0}{1.07} \quad \text{(6)}
\]

where the correction is 7%; the factor “2” takes into account fact that \( a \) is now the radius of circular membranes (for square membranes, \( a \) is the side of a square). This correction is confirmed on the calculated data of critical buckling:

\[
k_0^{cr} (\text{cir}) = 3.83, \quad k_0^{cr} (\text{sq}) = \frac{4\pi}{\sqrt{3}}, \quad \text{thus}
\]

\[
k_0^{cr} (\text{sq}) = 2k_0^{cr} (\text{cir}) \quad \text{(7)}
\]

In the absence of initial stress \( (k_0=0) \), and for small deflections \( (h/e=0.8) \), the coefficient \( C(v, h/e, k_0) \) is respectively calculated by FEM for both circular and square membranes:

\[
C(\text{cir})(v, 0, 0) = \frac{1.87}{1-v^2} \quad \text{and}
\]

\[
C(\text{sq})(v, 0, 0) = \frac{4C(\text{cir})}{1.23(1-v^2)} = \frac{6.1}{1-v^2}. \quad \text{(8)}
\]

The first equation corresponds to the analytic solution given by Timoshenko and Woinowsky-Krieger [23]. The correction factor for square membranes here is 23%. For strong deflections (until \( h/e \approx 7 \)), \( C \) decreases with \( h/e \) and the adjustment of FEM results by an analytic function is made for square membranes with:

\[
C(\text{sq}) \left( \frac{v}{e}, \frac{h}{e}, 0 \right) = \frac{6.1}{1-v^2} \left[ 1 - 0.47 \left( 1 - \exp -0.168 \frac{h}{e} \right) \right]. \quad \text{(9)}
\]

For bigger deflections \( (h/e \gg 1) \), \( C \) is constant: \( C(\text{sq}) = (3.23)/(1-v^2) \).

\( C \) is a function of \( k_0 \) for both compressive and tensile stress. Until the value of the limit stress of buckling the influence of \( k_0 \) on \( C \) is negligible. This approach permits the applicability of “point-wise” deflection technique to (100) silicon membranes of square geometry with PECVD-grown SiO\(_2\). Eq. (2), linking the applied load \( P \) at the center of membrane with the out-of-plane displacement \( h \), becomes:

\[
\frac{P}{h} = \frac{A}{g(k_0)} + BC \left( \frac{v}{e}, \frac{h}{e}, 0 \right) h^2 \quad \text{with}
\]

\[
A = \frac{D_{eff}}{\pi a^2} \quad \text{and} \quad B = \frac{E_{eff}e_{eff}}{a^2}. \quad \text{(10)}
\]

The zero of the linear function \( P/h = f(h^2) \) and the knowledge of \( D_{eff} \) permitted the determination of \( g(k_0) \) and \( k_0 \), and finally, the extraction of \( \sigma_{eff} \) and \( \sigma_1 \) (Eq. (3)). There are four following solutions: (a) \( P/Ah=1 \) with \( g(k_0)=1 \) in the absence of stress; (b) \( P/Ah<1 \) with \( 0<g(k_0)<1 \) in tensile situation; (c) \( 0<P/Ah<1 \) with \( g(k_0)>1 \) in the presence of compressive stress before buckling; and (d) \( P/Ah<0 \) with \( g(k_0)<0 \) in the presence of compressive stress of magnitude greater than the value of critical stress of buckling \( \sigma_0^{cr} \). The monitoring the function \( C \) (Eq. (9)) permits to extract \( E_{eff} \) and \( E_1 \) by determination of the slope of function \( P/h \) (Eq. (10)). When \( k_0^{cr}>k_0^{eff} \), the deflection generated by compressive stress can be bigger than membrane thickness \( (h_0=e) \). In this case, the tensile stress can appear when applying the “point-wise” deflection technique. Therefore, we determine experimentally the values of \( k \) in the range \( k \approx k_0 \).
When the membrane presents an initial bending $h_0$ due to the buckling, it is necessary to make a translation on the displacement $h$ [19]. Eq. (10) becomes:

$$\frac{P}{h + h_0} = \frac{A}{g(k)} + BC\left(\frac{h}{e} - 0\right)(h + h_0)^2$$

with

$$k^2(cir) = k_0^2(cir) - 9.7\left(\frac{h_0}{e} - 0.8\right)^2$$

where function $g(k)$ is obtained by combining interferometric data of the initial bending $h_0$ and linear representation of Eq. (11); (−) are a Mac Cawley brackets.

4. Results and discussion

4.1. Properties of SiO$_x$N$_y$ films

Experimental data of chemical composition are summarised in Table 2 where the film thickness, the atomic percent (at.%) of silicon, nitrogen, oxygen and hydrogen, and mass density of deposited films with various refractive indexes are given. Atomic concentrations were calculated from the layer thickness obtained in the analysis in units of atom/cm$^2$ [25]. Table 2 shows that the quantities of incorporated nitrogen increase (0–52%) with increasing refractive index, while the quantities of oxygen decrease (65–0%) with increasing refractive index. The silicon content (29–32%) is relatively independent of the refractive index. Hydrogen has particularly important effect on the mechanical and optical properties of deposited films: the atomic percentage increases rapidly (2–18.8%) with increasing refractive index. Solid line at Fig. 5 displays the atomic densities as a function of refractive index. The experimental data reveal a relatively linear increase in density with increasing refractive, accompanied by a jump for the sample b ($n = 1.517$). The oxide-like film (sample a, $n = 1.469$) has a density slightly upper than fused silica with 2.29 g/cm$^3$. The nitride-like film (sample f, $n = 1.815$) has the higher density with 2.70 g/cm$^3$. It will be shown later that denser films result in more compressive stress.

![Fig. 5. Atomic densities of SiO$_x$N$_y$ films and variation of etch rate versus the refractive index of samples (a–f).](image)

The variations of etch rate with the evolution of the SiO$_x$N$_y$ refractive index is represented by dashed line in Fig. 5. For the refractive index of 1.645 (sample d), the atomic percentages of oxygen and nitrogen are equivalent and then the atomic percentage of nitrogen becomes higher than the percentage of oxygen. The etch rate evolution follows this tendency, increasing from sample a to sample d ($n = 1.469–1.645$) where the etch rate is maximal, and then decreasing with increasing refractive index. We can conclude that the oxygen/nitrogen content influences the etch rate rather than hydrogen content.

4.2. Mechanical properties of SiO$_x$N$_y$ membranes

To measure Young’s modulus $E_s$ of the substrate in the absence of initial stress, we first investigated the behavior of (100) silicon membranes ($a = 3.6$ mm, $e_s = 15$ μm) without SiO$_x$N$_y$ layer. A deflection of 10 μm was produced by the nanoindenter on a series of membranes, as shown in Fig. 6 illustrating the Eq. (11) when $h_0 = 0$. The magnitude of stress was determined of these membranes is low: $\sigma_s = \frac{E_s}{1 + \nu} = 1$, 4 and −6 MPa, and the measured Young’s ($E_s = 130 \pm 4$ GPa when $\nu = 0.28$) is perfectly matched with calculated one ($E_s = E_e = 129$ GPa). For $\sigma_s = 0$ and $E_s = 129$ GPa, we can define $\sigma_f$ in bimorph situation as:

$$\sigma_f = \sigma_{eff}\left(1 + \frac{e_s}{e_f}\right).$$

In the second step, we investigated the bimorph membranes with evaluation of Young’s modulus. Fig. 7 shows

![Fig. 6. Plot of the function $P/h$ versus $h^2$ in case of Si membrane.](image)

![Fig. 7. Young’s modulus versus the refractive index of SiO$_x$N$_y$ film.](image)
the quasi linear increase of Young’s modulus as a function of refractive index of SiO$_N$$_y$ films. We have seen that the refractive index $n$ increases linearly following the changes in the concentration of oxygen and nitrogen, according to the modifications of gas flow ratio $R=N_2O/(N_2O+NH_3)$. The evolution of $E_r$ as a function of $n$ is in agreement with this linear law. For oxide-like films (series B, $n=1.51$) the Young’s modulus (78 GPa) is close to the modulus of the thermal oxide (70 GPa), while for the nitride-like film (series F, $n=1.81$), the Young’s modulus (148 GPa) is close to the modulus of pure Si$_3$N$_4$ [26]. The measured values are in good agreement with the literature data [26,27].

By analogy with the behaviour of Young’s modulus, both hardness values $H_f$ (value of $H$ for $h \to 0$) and $H_\infty$ (value of $H$ for $h=900$ nm) increase linearly with the refractive index of SiO$_N$$_y$ films, as shown in Fig. 8.

Finally, the initial deflection $h_0$ of membranes was measured by interferometry. Fig. 9 shows the distribution of out-of-plane displacement $h_0$ versus the refractive index of the SiO$_N$$_y$ films for series B, C, D, E and F. The curve presents a minimal bending ($h_0 \approx 0$) for the refractive index $n=1.58$ (series C). After that optimum, the deflection is increasing with the increase of the refractive index.

4.3. Comparison of experimental results with theoretical analysis

The representation of Eq. (11) in case of bimorph membranes is given in Fig. 10, where three categories of samples are shown (two membranes by class of series C, E and four for the F). The knowledge of $h_0$, $E_r$, $v_r$, $E_t$ and $v_f$ permits to follow the variation of $\sigma_r$ as a function of refractive index. In addition, the values of $E_{eff}$ can be estimated and compared with the theoretical calculation. For each class of samples with same refractive index, the straight lines of Fig. 10 crosses the same origin on the vertical axis, even presenting different slopes due to the dispersion of $h_0$ values. Consequently, the function $g(k)$ has the same value for each category of sample with same $\sigma_{eff}$. The standard deviation of $\sigma_r$ is low. On the other hand, the variation of individual slopes depends strongly from the values of $h_0$, introducing a significant dispersion of $E_{eff}$ results. The comparison of experimental and theoretical results is given in Table 3.

The distribution of residual stress $\sigma_f$ versus the refractive index of SiO$_N$$_y$ thin films is shown in Fig. 11. This graph gives the fitting curve for averaged experimental data. In Table 3, deflections $h_0$ with sign $\pm$ means a stress of tensile nature in the film of the buckled membrane. This is

![Fig. 8. Hardness $H_f$ ($h \to 0$) and $H_\infty$ ($h \to 900$ nm) versus the refractive index of SiO$_N$$_y$ film.](image)

![Fig. 9. Initial deflections $h_0$ versus the refractive index of SiO$_N$$_y$ film.](image)

![Fig. 10. Plot of the function $P(h+h_0)$ versus $f(h+h_0)^2$ in the case of bimorph membranes.](image)

![Table 3 Values of $h_0$, $g(k)$, $\sigma_r$, $E_{eff}$测 and $\sigma_{eff}$测 versus the refractive index of SiO$_N$$_y$ film.](image)

![Comparison of theoretical and the experimental values of $E_{eff}$.](image)
due to the passage of certain membranes from the compressive situation (convex membrane) to the tensile one (concave membrane) during the sample removing from PECVD furnace (passage from vacuum to the ambient air). The term \( \alpha^* \) describes the membranes whose border was clamped mechanically during the measurement session. Consequently, a very low tensile stress (\( \sigma_t = +16.0 \text{ MPa} \)) is measured (instead of compressive one in the range of \( -130 \text{ MPa} \)). This demonstrates clearly the strong consequences of mechanical clamping of the sample on the stress evaluation. When the refractive index is increasing, the minimal stress is obtained for \( n = 1.58 \) with \( \sigma_t = -100 \text{ MPa} \), as shown in Fig. 11. We have seen that around this refractive index (sample C), the film density is lower. For nitride-like films, the stress becomes more compressive, increasing with the increase of refractive index. This attains \( -400 \text{ MPa} \) around \( n = 1.81 \). On this graph, the evolution of the critical film stress of buckling is plotted:

\[
\sigma_t^c \approx \sigma_{\text{eff}}^c \left(1 + \frac{e_s}{e_t}\right) \quad \text{with} \quad \sigma_{\text{eff}}^c = \frac{16\pi^2}{3} \frac{D_{\text{eff}}}{e_{\text{eff}} \alpha^2}.
\]

For the series C (\( n = 1.58 \)) where \( g(k_0) < 0 \), the measured stress is lower than the critical stress of buckling (\( \sigma_t < \sigma_t^c \)). This is accompanied by a very low initial deflection \( h_0 \). Other samples lead to the buckling of bimorph structures with \( g(k_0) > 0 \). In conclusion, the optimal situation of SiO\(_x\)N\(_y\) deposition when the stress is of compressive nature and the membrane in the state of prebuckling is obtained for \( n = 1.58 \) (series C).

5. Concluding remarks

In this paper, an optomechanical approach of characterisation the mechanical performances of SiO\(_x\)N\(_y\) films has been demonstrated for the particular case of compressively prestressed silicon membranes by deposition of SiO\(_x\)N\(_y\) films. All SiO\(_y\)N\(_y\) films that we fabricated by PECVD process, operating at the temperature of 350 °C and obtained with low hydrogen content, are of compressive nature. Danaie et al. [28] reported that SiO\(_x\)N\(_y\) membranes with tensile stress can also be fabricated at low-frequency PECVD reactor when the thin film is deposited with higher hydrogen content and annealed after deposition to reduce the hydrogen content.

Table 4 shows an overview of measured values from the literature compared with the results of our contribution. SiO\(_x\)N\(_y\) layers are obtained from PECVD reactors operating with SiH\(_4\)-NH\(_3\)-N\(_2\)O gas mixture. We can see a clear tendency concerning the magnitude of Young’s modulus. Thus, Danaie et al. [28] measured the Young's modulus between 86 and 149 GPa within the range of refractive indices between 1.48 and 1.9, while Kramer and Paul [18] obtained 150 GPa for a refractive index above 2. This is in agreement with our estimation of the Young’s modulus covering the range from 78 to 147 GPa for refractive indices within 1.51–1.81. The direct comparison is less evident concerning the residual stress magnitude because of the large number of processing parameters and the use of different PECVD reactors, although these estimations were made from measurements on membranes or on wafers. In the meantime, certain trends can be seen from the stress data. All deposited films were found to have a compressive stress. Our results are in agreement with estimation reported by Denisse et al. [29], demonstrating a similar level of compressive stress (240 to 310 versus 90 to 400 MPa for similar range of refractive index 1.5 to 1.7 versus 1.49 to 1.81). These results are confirmed by Kramer and Paul [18] that measured the compressive stress of 320 MPa for a refractive index above 2. Danaie et al. [28] and Classen et al. [30] found higher compressive stress for refractive indices of about 1.9. In this work, higher SiH\(_4\) flow (57 versus 7 sccm) resulted in higher stress value. Consequently, all these results would also appear to be in agreement.

The observation that PECVD SiO\(_x\)N\(_y\) films can be deposited with a low compressive stress while having good

| Ref. | Nature of deposition | Stress [MPa] | Young’s modulus [GPa] | Refractive index | Pressure [mbar] | Temperature [°C] |
|------|----------------------|-------------|----------------------|-----------------|----------------|-----------------|
| Danaie et al. [28] | 400 nm on Si wafer | −330 to −1050 | 86–149 | 1.48–1.9 | 500 | 200–300 |
| Kramer and Paul [18] | 1.9 μm on Si membrane | −320 | 150 | 1.9–2.0 | – | 300 |
| Denisse et al. [29] | on Si wafer | −240 to −310 | – | 1.5–1.7 | 1.45 | 380 |
| Classen et al. [30] | Si wafer | −170 to −780 | – | 1.51–1.91 | 0.65 | 300 |
| Allaert et al. [31] | 100 nm on Si wafer | −180 | 1.85 | 3.6 | 220–315 |
| This work | 2 μm on Si membrane | −90 to −400 | 78–147 | 1.51–1.81 | 0.14 | 350 |

Fig. 11. Distribution of residual stress versus the refractive index of the SiO\(_x\)N\(_y\) film.
control of refractive index makes these layers attractive candidates for application as core layer in channel optical waveguide in MOEMS devices.

Acknowledgements

This work is supported by Growth Programme of the European Union (contract G1RD-CT-2000-00261). L. Nieradko acknowledges the support from Marie Curie Individual Fellowship (contract HPMF-CT-2001-01200). The authors thank Pascal Blind from the Centre de Transfert des Microtechnologies (Besançon, France) for his help in fabrication of samples.

References

[1] C. Gorecki, in: P. Rai-Choudhury (Ed.), MEMS and MOEMS—Technology and Applications, SPIE Press, Bellingham, 2000, p. 209.

[2] W.N. Sharpe Jr., B. Yuan, R. Vaidyanathan, in: M.T. Postek, C. Friedrich (Eds.), Micromachining and Microfabrication ’96, Microlithography and Metrology in Micromachining II, Austin, TX, U.S.A., October 14–15, 1996, Proceedings of the SPIE, vol. 2880, International Society for Optical Engineering, Bellingham, WA, 1996, p. 78.

[3] H. Ogawa, K. Suzuki, S. Kaneko, Y. Nakano, Y. Ishikawa, T. Kitahara, The 10th Annual International Workshop on MEMS, Nagoya, Japan, January 26–30, 1997, Proceedings of the IEEE 97CH36021, 1997, p. 430.

[4] P.J. French, P.M. Sarro, R. Mallet, E.J.M. Fakkeldij, R.F. Wollenbuttel, Sens. Actuators, A, Phys. 58 (1997) 149.

[5] W. Osten, W. Juptner, S. Seebacher, T. Baumbach, in: C. Gorecki (Ed.), Industrial Lasers and Inspection: Microsystems Metrology and Inspection, Munich, Germany, June 14–18, 1999, Proceedings of the SPIE, vol. 3825, International Society for Optical Engineering, Bellingham, WA, 1999, p. 152.

[6] S. Seebacher, W. Osten, T. Baumbach, W. Juptner, Opt. Lasers Eng. 36 (2001) 103.

[7] S. Petitgrand, R. Yahiaoui, K. Danaie, A. Bosseboeuf, J.P. Gilles, Opt. Lasers Eng. 136 (2000) 77.

[8] P. Aswendi, R. Hofling, K. Hiller, in: C. Gorecki (Ed.), Industrial Lasers and Inspection: Microsystems Metrology and Inspection, Munich, Germany, June 14–18, 1999, Proceedings of the SPIE, vol. 3825, International Society for Optical Engineering, Bellingham, WA, 1999, p. 165.

[9] V. Petrov, B. Lau, in: M. Kujawińska, W. Osten (Eds.), Industrial Lasers and Inspection: Optical Measurement Systems for Industrial Inspection, Munich, Germany, June 14–18, 1999, Proceedings of the SPIE, vol. 3824, International Society for Optical Engineering, Bellingham, WA, 1999, pp. 262–273.

[10] H. Bezzaoui, E. Voges, Sens. Actuators, A, Phys. 29 (1991) 219.

[11] E. Bonnotte, C. Gorecki, H. Toshiyoshi, H. Kawakatsu, H. Fujita, K. Wörhoff, K. Hashimoto, J. Lightwave Technol. 17 (1999) 35.

[12] C. Poilane, P. Delobelle, L. Lexcellent, S. Hayashi, H. Tobushi, Thin Solid Films 379 (2000) 156.

[13] C. Gorecki, A. Sabac, M. Jóźwik, S.S. Lee, in: A.K. Asundi, W. Osten, V.K. Varadan (Eds.), ISPA-Photonics and Applications, Advanced Photonics Sensors and Applications II, November 26–30, 2001, Singapore, Proceeding of the SPIE, vol. 4596, International Society for Optical Engineering, Bellingham, WA, 2001, p. 9.

[14] W.C. Oliver, G.M. Pharr, J. Mater. Res. 7 (1992) 1564.

[15] G.M. Pharr, W.C. Oliver, J. Mater. Res. 7 (1992) 613.

[16] M. Jóźwik, C. Gorecki, A. Sabac, P. Delobelle, M. Kujawińska, Opt. Lasers Eng. 41 (2003) 703.

[17] M. Kujawińska, Automatic fringe pattern analysis, in: K. Patorski (Ed.), Handbook of the Moiré Fringe Technique, Elsevier, Amsterdam, 1993, p. 339.

[18] T. Kramer, O. Paul, J. Micromechanics Microengineering 12 (2002) 475.

[19] M.K. Small, W.D. Nix, J. Mater. Res. 7 (1992) 1553.

[20] O. Tabata, K. Kawabata, S. Sugiyama, I. Igarashi, Sens. Actuators 20 (1989) 135.

[21] E. Bonotte, P. Delobelle, L. Bornier, B. Troland, G.J. Tribillon, J. Mater. Res. 12 (1997) 2234.

[22] S. Hong, T.P. Weihs, T.C. Bravman, W.D. Nix, J. Electr. Mater. 19 (1990) 903.

[23] S. Timoshenko, S. Woinowsky-Krieger, in: Ch. Beranger (Ed.), Théorie des Plaques et Coques, Libraire Polytechnique, Paris, 1961, in French.

[24] D. Maier-Sneider, J. Maibach, E. Obermeier, J. Micromech. Syst. 4 (1995) 238.

[25] L.G. Parrat, Phys. Rev. 95 (1954) 359.

[26] K. Danaie, DSc Thesis, Université Pierre et Marie Curie (Paris VI), France, 2002, in French.

[27] J.J. Vlassak, W.D. Nix, J. Mech. Phys. Solids 42 (1994) 1223.

[28] K. Danaie, A. Bosseboeuf, C. Clerc, C. Gousset, G. Julie, Sens. Actuators, A, Phys. 99 (2002) 78.

[29] C.M.M. Denisse, K.Z. Troost, J.B. Oude Elferink, F.H.P.M. Harken, M. Hendriks, J. Appl. Phys. 60 (1986) 2536.

[30] W.A. Classen, H.A.J. V. D. Pol, A.H. Goemans, A.E.T. Kuiper, J. Electrochem. Soc. 33 (1986) 1458.

[31] K. Allaert, A. Van Calster, H. Loos, A. Lequesne, J. Electrochem. Soc. 137 (1985) 1763.