DEVELOPMENT OF THE BULGE TEST EQUIPMENT FOR MEASURING MECHANICAL PROPERTIES OF THIN FILMS

HOLZER Jakub[1], PIKÁLEK Tomáš[2], BUCHTA Zdeněk[2], LAZAR Josef[2], TINOCO Hector A.[3], CHLUPOVÁ Alice[1], KRUML Tomáš[1]

[1]Institute of Physics of Materials of the CAS, Brno, Czech Republic, EU
[2]Institute of Scientific Instruments of the CAS, Brno, Czech Republic, EU
[3]Universidad Autónoma de Manizales, Manizales, Colombia

Abstract

The bulge test apparatus designed for the measurement of mechanical material properties of thin films was constructed and tested. The principle of the test is to apply pressure on a free-standing membrane, to measure the membrane shape and to analyse the results. Commercially available silicon nitride (Si_3N_4) thin films were used for the testing. It is shown that interferometric set-up designed and assembled for the apparatus enables precise determination of 3D shape of the whole membrane, which allows more precise determination of materials parameters compared to measurement of the height of the center of the membrane only. Fit of an analytical formula gives values of Young modulus and residual stress with very good agreement with the literature data. Moreover, FEM model of the bulged membrane was developed. The main aim of the effort is to enable measurement of plastic properties of a thin film of interest, that will be deposited on the Si_3N_4 membrane with known properties and bulge test will be performed on the bilayer specimen. Subsequently, the material properties of the thin film will be obtained using FEM analysis.

Keywords: Thin films, bulge test, mechanical properties

1. INTRODUCTION

Thin films have been used in many industrial and medical applications such as microelectromechanical and nanoelectromechanical systems and medical sensors throughout recent years and their importance is still growing [1,2]. Due to increased applications of thin films, new techniques for determining mechanical properties are needed. Behavior of thin film materials undergoing stress and deformation are disparate from bulk materials, therefore reliable testing methods had to be developed. Bulge test aside, several techniques have been introduced including nanoindentation [3], nano-scale tensile tests [4] and beam bending tests [5]. Nanoindentation method is widely used and suitable for measuring hardness and elastic properties, such as Young's modulus and Poisson's ratio, however, its ability to determine plastic properties is questionable. All of these different approaches meet with several difficulties, for instance sample preparation and data analysis [6,7].

The bulge test technique consists of applying pressure on a free-standing membrane and measuring and analysing the membrane shape. It was originally proposed and used by Beams in 1959 [8]. While the principle of this technique is relatively simple, two difficulties occur: i) fabrication and manipulation with thin membranes, ii) analysis of results, i.e. deriving the relationship between pressure and membrane shape. Many methods of manufacturing the free-standing membranes could be found in the literature. Probably the most precise one takes advantage from the micromachinig techniques used in microelectronic industry [6]. Free standing membranes are produced by photolithography and selective etching of silicon substrate with thin film deposited on them. Due to etching dependence on crystallographic planes, it is only possible to make square and rectangular windows. Commercial Si_3N_4 membranes deposited on silicon wafer are available. Several authors used these membranes for determining properties of thin films following this procedure: the properties of commercial membranes are precisely measured, another thin film of interest is deposited on it and bulge test is performed on the bilayer specimen [9]. Subsequently, the material properties of the thin film are obtained.

In this paper, we report our effort in further developing of this technique.

2. EXPERIMENT

2.1. Specimens

Si frames with square and rectangular windows with deposited Si_3N_4 thin films were fabricated by the Norcada company. Results presented here were obtained on 2 x 2 mm square membrane (**Figure 1**). The thickness of the membrane was measured using SEM Lyra3 Tescan on a broken specimen as 590 nm. Clamping membranes to experimental device proved to be challenge mainly due to fragility of the thin membrane. Pressing silicon window between two seals led often to membrane rupture or destruction of the silicon wafer itself. Therefore, another method was developed. Instead of clamping silicon wafer between two seals, it was glued using cyanoacrylate onto PMMA support with hole in the middle. This way, no external stress is applied on the membrane.

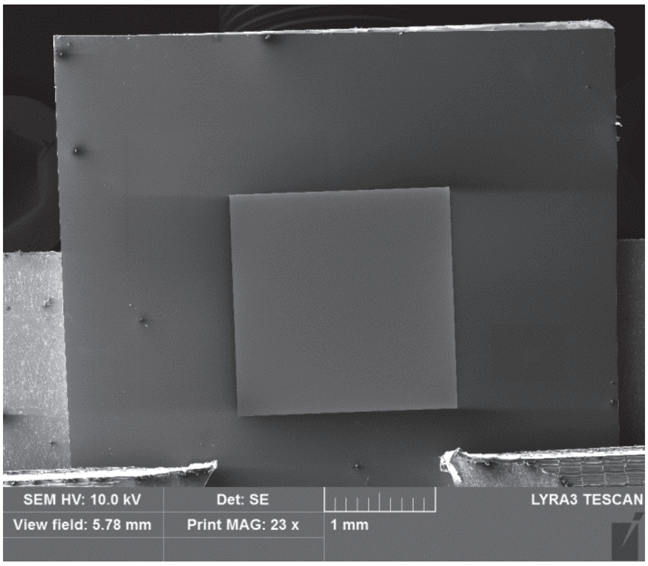

Figure 1 Si frame with Si_3N_4 membrane

2.2. Bulge test apparatus

The bulge test apparatus was constructed as schematized in **Figure 2**. The differential pressure is applied using an industrial grade piston that presses the air by computer-controlled syringe pump. Pressure is measured by the low pressure transmitter with maximal error 0.5% connected to computer. The membrane shape is measured using an interferometric system (**Figure 3**). The light source for the Michelson-type interferometer is a 633 nm fiber-coupled HeNe laser. The diverging beam from the fiber is collimated into diameter 10 mm using a doublet lens. The beam is then split into a measuring beam that reflects off the measured sample and a reference beam that reflects off a reference mirror with high surface flatness λ/10. The measuring beam then interferes with the planar reference beam at the output of the interferometer forming interference fringes that are projected onto the camera sensor using a camera lens (Carl Zeiss Pancolar 50mm f/1.8). The captured interferograms on each camera pixel are then used to calculate the change of the z position of the membrane during the experiment with resolution better than half of the laser wavelength. Since the beam diameter is larger than the

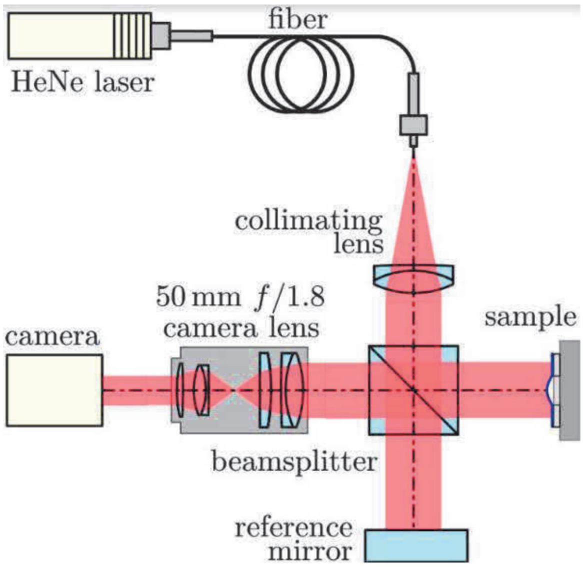

Figure 2 Schematics of the bulge test apparatus

size of the membrane and the camera is used to capture the interference fringes, the entire membrane shape can be measured. Interference fringes of bulged membrane are visible in **Figure 4**.

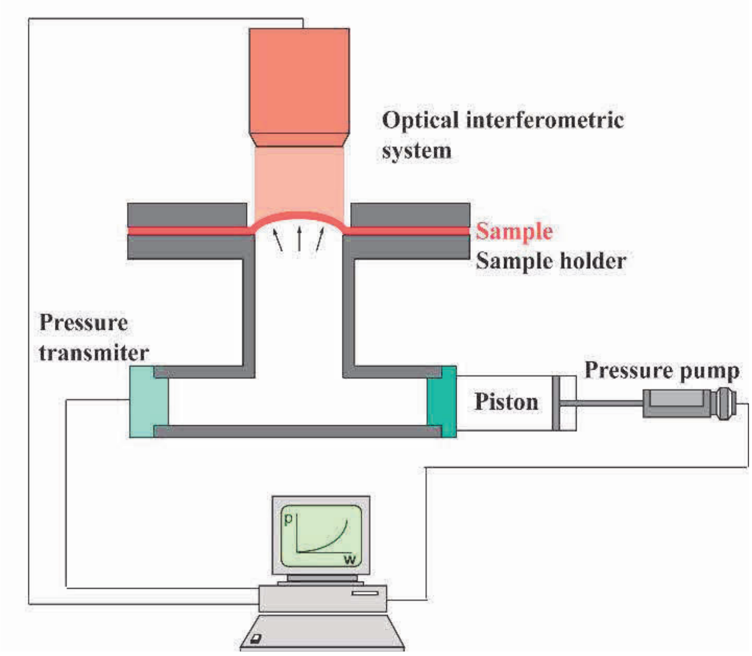

Figure 3 Interferometer setup

The membranes were pressured from 0 to 10kPa and back to unpressured state. More than two thousands measurements of the membrane shape were done in one test. Number of points for which the z position was evaluated is 152x152. 3D reconstruction of the membrane shape was made in MATLAB software. The bulge test was performed with positive and negative pressure, however, no difference in the membrane shape was observed.

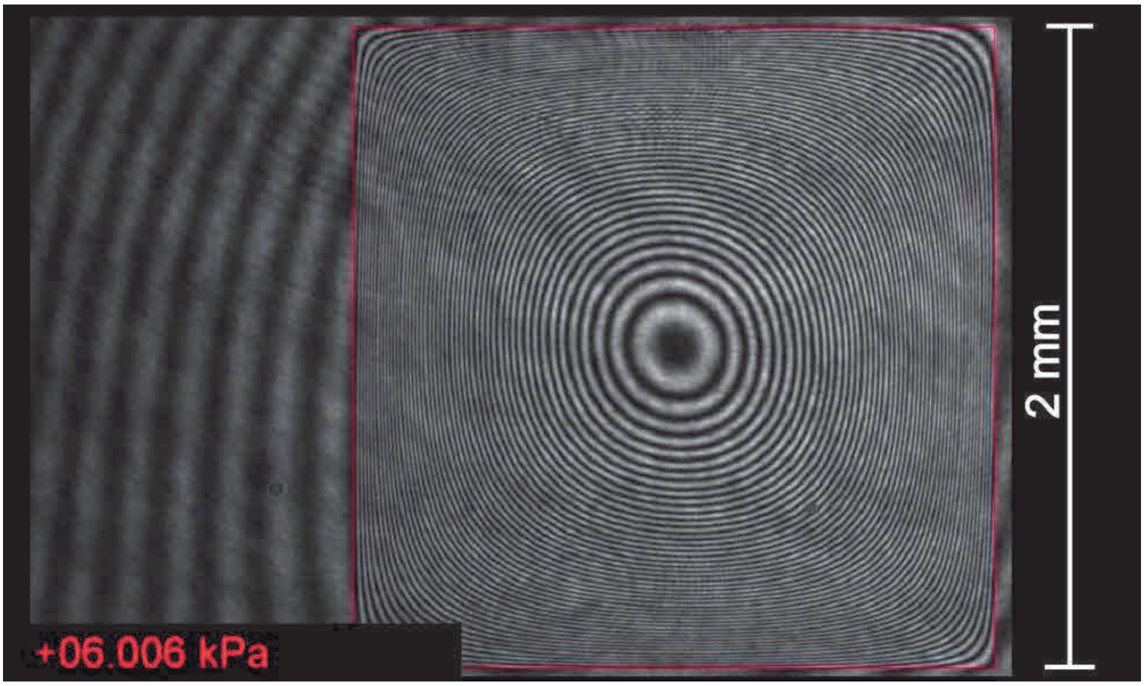

Figure 4 Interferometric image of the membrane at 6 kPa pressure

3. DATA ANALYSIS

3.1. Analytical model

By applying pressure to one side of the membrane and measuring the shape of bulge, we are able to construct pressure-deflection relationship which is then used as data to fit analytical equations describing theoretical relation between height of the center of the membrane (*w*) and pressure (*p*). Following equation was used [9]:

$$p = C_1 \frac{\sigma_0 t w}{a^2} + C_2 \frac{E t w^3}{a^4 (1 - \nu)} \tag{1}$$

where *a* is the half-length of membrane window, *t* the thickness of film, σ_0 the residual tensile stress which appears in the film during the film preparation due to different thermal coefficient of the film and Si substrate, *E* the Young's modulus and ν the Poisson's ratio. C_1 is constant which depends on membrane geometry and C_2 also depends on membrane geometry, but it is a weak function of the Poisson's ratio as well. The first part of the equation describes the linear effect of residual stress σ_0 in the membrane, whereas second term is linked to stretching of the membrane under uniform pressure and contains elastic constants of given material [10].

Eq. 1 was fitted to experimental data. The result is shown in **Figure 5**. The overlap between experimental curve and the fit is very good. Assuming Poisson ratio as 0.27 [9], Young modulus derived from the fit is 207 GPa, residual tensile stress is 163 MPa. Other parameters used in Eq.1 were: C_1 = 3.393, C_2 = 1.83 [9], *t* = 590 nm and *a* = 1mm.

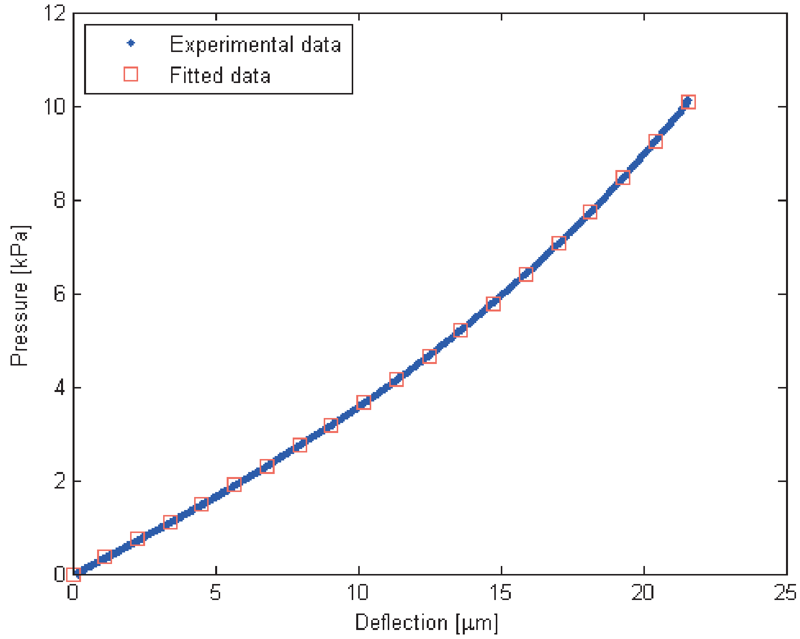

Figure 5 Pressure - deflection curve for 2x2 mm membrane center and the fit of Eq. 1. Experimental data point are in blue (25000 data points), the fit is represented by red squares.

3.2. Finite element model

For a square membrane with fixed borders subjected to pressure, Eq. 1 solves the displacement of the membrane center along the z-axis. Since the interferometric setup provides quite precise information of the whole membrane shape, more detailed analysis can be achieved. Therefore, an effort to obtain material parameters using finite element analysis which takes into account the whole membrane is ongoing. To approximate the displacement field determined experimentally, a finite element analysis is carried out in

ANSYS software. The numerical model considers geometric nonlinearity, i.e. large deformations are included in the model. **Figure 6** shows a comparison between experimentally determined (**Figure 6a**) and numerically calculated (**Figure 6b**) shapes of the membrane at 10 kPa pressure. Input material parameters (Young's modulus 207 GPa and residual stress 163 MPa) were taken from results of the analytical model. The difference between the experiment and the model is shown in **Figure 6c**.

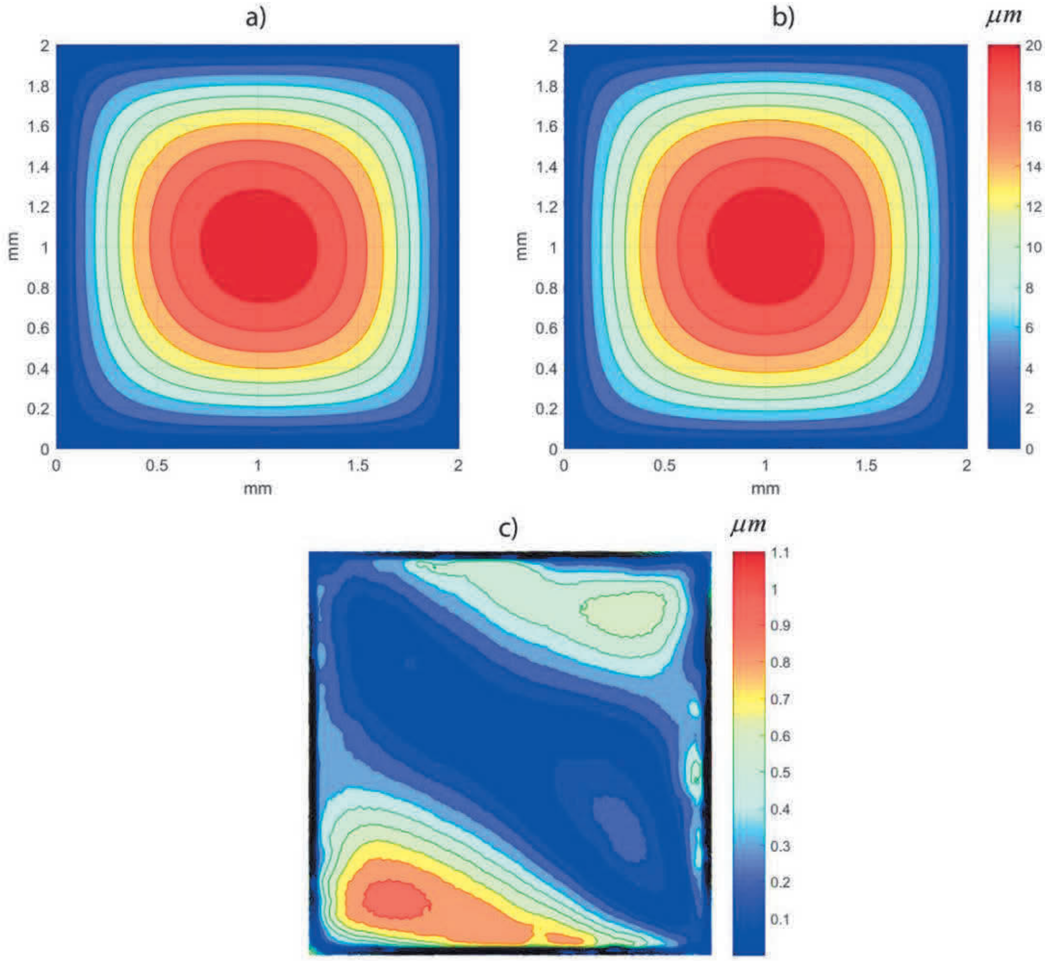

Figure 6 3D shape of the membrane, z position is described by color coding. a) Experimental shape of the membrane derived from the interferometric measurement, b) calculated shape of the membrane by FEM, c) difference between the two shapes; p = 10kPa.

4. DISCUSSION

Pressurizing apparatus and optical setup proved to be suitable for the purpose. Some difficulties appeared with clamping of specimens. Uneven deformation of the Si frame appeared during tightening which led to rupture of the membrane. Gluing the specimen on PMMA support eliminated the problem. The first results calculated by the analytical model (Young's modulus 207 GPa and residual stress 163 MPa) match results given by other research teams quite well. Giving some examples from the literature in Young's modulus [GPa]/residual stress [MPa] order: 225/227 [9], 255/217 [11], 217/411 [12], 222/- [13]. The manufacturer of the membranes used in this study reports residual stress being less than 250 MPa, which again corresponds with our outcome. Finite elements analysis was applied and proved to reproduce the experimental membrane shape quite well, when non-linear geometric parameters are used. Further study using numerical simulation will be carried out with the aim to expose more details about material parameters and behavior. Reproducibility of the measurement and its precision must be further analysed. Interferometric setup designed for the apparatus supplies quite precise information about the membrane shape; however, some differences between experimental and simulated shapes appears close to two corners of the membrane (**Figure 6c**). Possible explanation may lay in non-uniformity of membrane (e.g. variation in thickness), in aberrations of lenses or possible necessity of more realistic numerical model. Further investigation will be carried out.

5. CONCLUSIONS

- Apparatus for the bulge test measurement was successfully constructed;
- Interferometric measurement enables precise determination of the shape of whole membrane;
- Young's modulus 207 GPa and residual stress 167 MPa were determined using analytic formula in a good agreement with the literature;
- FEM model is under development with the aim to obtain more material data and enable elasto-plastic analysis of bilayer film.

ACKNOWLEDGEMENTS

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