APPLIED MECHANICS 2018
April 9 - 11, 2018, Myslovice, Czech republic

ESTIMATION OF MECHANICAL PARAMETERS OF THIN FILMS USING FINITE ELEMENT ANALYSIS

H. A. Tinoco*[1,2,3], J. Holzer[2], T. Pikálek[4], Z. Buchta[4], J. Lazar[4], A. Chlupová[2], T. Kruml[2], P. Hutař[2]

Abstract: This study shows a methodology to estimate mechanical parameters of thin films by means of a bulge test and a numerical approach. The methodology is based on the combination of finite element analysis with a classical analytical method. Finite element modelling was conducted for monolayer (Si3N4) membranes of 2x2mm with the aim to approximate both the load-deflection curves experimentally measured and the classical load-deflection analytical model. Error functions were constructed and minimized to delimit a coupled solution space between Young's modulus and Poison's ratio. In a traditional bulge test analysis only one of the elastic properties can be determined due to that there is not unique solution in the estimations of these parameters. However, both elastic parameters were determined through the proposed numerical procedure which compares the deformed surfaces for a specific set of optimal elastic parameters computed. Results show that the estimated elastic properties agree with corresponding values determined by other methods in the literature.

Keywords: Bulge test, thin film, finite element analysis, elastic properties

1 Introduction

Thin films are used in several engineering fields especially those related to microelectronics (ultra large scale integrated circuits), microelectromechanical systems (MEMS), nano-devices, coating applications, biomedical devices among others [1]. Integrated functionality has forced the processing of these structures to micro and nanometer scales. In this way, thin films require controlled processes and robust instrumentation to achieve the geometric conditions required for each application. However, the control of its mechanical behaviour presents additional challenges if intrinsic mechanical properties of thin films are unknown since it is not a trivial task to identify it till this time. Different experimental techniques have been developed to estimate mechanical properties of thin films, such as indentation, diffraction-based techniques, Raman spectroscopy, deflection techniques among others [2-3]. In contrast to the techniques mentioned above, a bulge test (deflection technique) can be highlighted and in comparison, with the other methods it presents several advantages that are mentioned and discussed in [3].

The bulge test technique consists of applying pressure on a membrane for measuring the displacement field [4-5]. Experimental load-deflection curve was analysed using different techniques to determine the elastic parameters that satisfy the measurements, in which we can point out fitting techniques and finite element analysis [6-8]. These techniques assume that the elastic parameters are decoupled or these are mechanically independent since in some cases, Poisson's ratio is fixed to obtain a solution for Young's modulus.

[1] *CEITEC-Central European Institute of Technology, Brno University of technology, Technická 10, 616 00 Brno, Czech Republic*
[2] *Institute of Physics of Materials, Czech Academy of Sciences, Žižkova 22, 616 62, Brno, Czech Republic*
[3] *Experimental and Computational Mechanics Laboratory, Universidad Autónoma de Manizales. Antigua Estación del Ferrocarril, Edificio Sacatín C.P. 170001. Manizales, Colombia*
[4] *Institute of Scientific Instruments, Czech Academy of Sciences, Královopolská 147, 616 64, Brno, Czech Republic*

This paper shows that the coupling conditions of Young's modulus and Poisson's ratio can be used for determining both elastic parameters combining finite element analysis with the classical identification method.

2 Materials and Methods

2.1 Experimental setup

A bulge test apparatus was constructed for thin films testing as shown in Figure 1a. The differential pressure is applied using an industrial grade piston that presses the air by a computer-controlled syringe pump. The pressure is measured by a low-pressure transmitter with maximal error 0.02% FS which is connected to a data acquisition system. The shape of the membrane is captured by an interferometric system (Twyman-Green-type interferometer) in which the light source is composed by a fiber-coupled HeNe laser with wave-length of 633 nm. The beam is 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 (Nikon 50mm f/1.4 NIKKOR G). Interference signals captured on each camera pixel are then used to determine the displacement field at the z position which is normal to the surface of the thin film. Number of points for which the z position was evaluated is dependent on the size, but usually exceeds 40 000. In a left part of Figure 1b, the scheme of the setup is shown. Right part of Figure 1b presents interference pattern corresponding to the tested membrane deformation. The device is also equipped with sensors to measure the ambient temperature, pressure and humidity required for air refractive index calculation. Detailed information about the experimental setup can be found in [9].

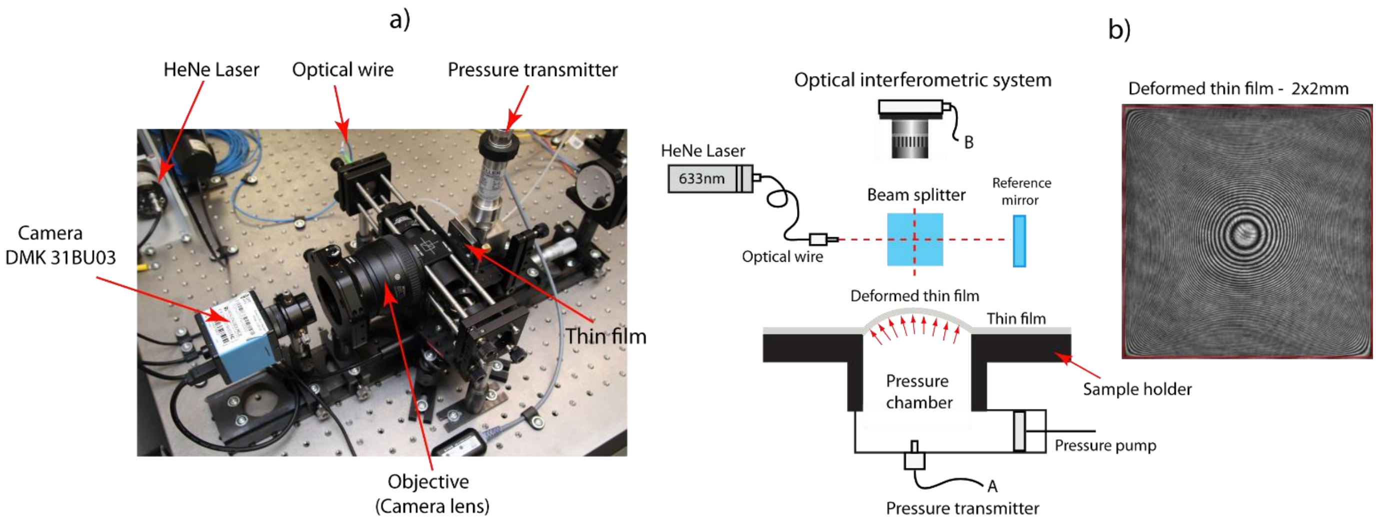

Figure 1: a. Experimental setup of bulge test. **b.** Scheme of the experimental setup and image of the membrane.

2.2 Classical analytical equation for estimation of mechanical parameters

Let's considers a rectangular thin film $2a \times 2b$ pre-stressed by σ_r and made of an isotropic elastic material that satisfies a linear stress–strain relationship. Under pressure conditions P, the shape of the thin film is defined by the surface deformation. In those conditions, [6] reported a classical analytical equation that relates the maximum deflection w_0 and pressure P as follows

$$P = C_1 \frac{\sigma_r t w_0}{a^2} + C_2(v) \frac{E t w_0^3}{a^4}, \tag{1}$$

where c_1 and $c_2(v)$ are constants that depend on geometric and material parameters, different models and numerical estimations have been proposed for both constants as described by [5], [7-8]. σ_r represents the residual stress, t is the thickness and E Young's modulus. In real applications, experimental data obtained for w_0 and P are fitted with the aim to determine σ_r, E and Poisson's ratio . However, the values of c_1 and c_2 are assumed according to the chosen approximation techniques as can be seen in [8], [10]. In a practical sense, these constants should take fix values but those depend on the techniques developed for each problem which in turn affect the identification process of properties. The coupling of the Young's modulus and Poisson's ratio have been neglected in the load-deflection analysis since one of the parameters should be fixed to estimate one of the elastic properties. To overcome these challenges, the combination of the analytical model with finite element analysis shows that an estimation of both elastic parameters can be carried out, as it will be described in the next sections.

2.3 Numerical approach for determining elastic properties

To determine Young's modulus and Poisson's ratio a sequential numerical procedure is presented in Figure 2 by means of a flow diagram. It consists in a set of 10 steps that permit to obtain both parameters combining finite element analysis with the classical analytical solution (see Equation 1).

Initially, a finite element model with the required geometric dimensions should be performed including its boundary conditions which are considered clamped in the external domain of the thin film as illustrated both in Figure 1b and Figure 2.

The bulge test modelling by finite element analysis is a well-known practice for many years ago (see [6-10]) since the geometries are very simplified and in practice the numerical analysis is not a complex engineering task considering that mechanical parameters are known. However, for the analysis it is important to take into account large deformations since the thicknesses are very thin (nanometric scale). Large deformations mean that the stiffness changes with the level of input load.

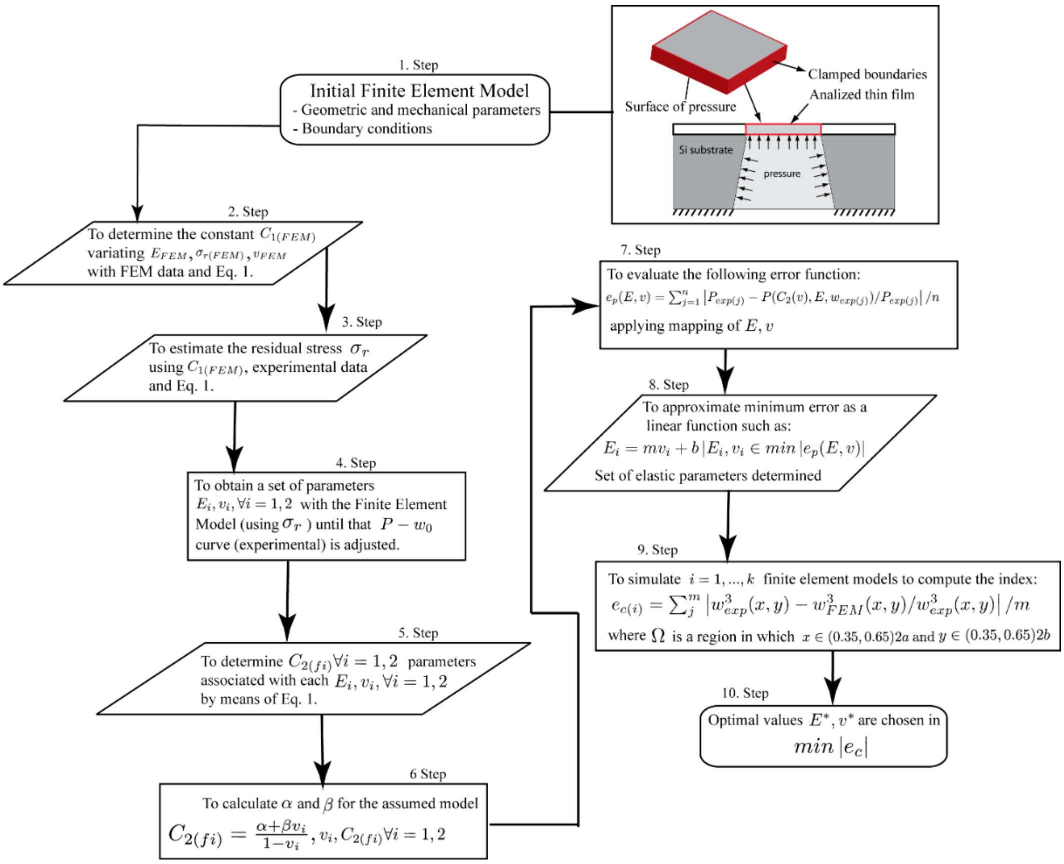

Figure 2: Sequential procedure for estimating Young's modulus and Poisson's ratio.

Following the proposed method, first three steps deal with the estimation of C_1 and σ_r parameters from the finite element model and experimental data. C_1 is dependent on the residual stress σ_r as described in [3]. Therefore, if with the finite element model a set of output data m is created with input parameters known ($E_j, \sigma_{rj}, v_j \forall j = 1, 2, ..m$), it is possible to determine C_1 adjusting those output data in Equation 1. Posteriorly, C_1 is used to compute the residual stress σ_r using the experimental data for any Young's modulus chosen in Equation 1. We suggest to choose a value close to materials with similar mechanical characteristics since for the true solution is an initial value.

In the steps 4, 5 and 6 the main objective is to establish a model for $C_2(v)$ constant only from the simulations. According to analytical solutions described by [6-10], $C_2(v) = \alpha + \beta v / 1 - v$ function is presented here, considering that different values of α, β have been determined with numerical and analytical approximations as summarized by [3]. In our case, we propose numerical estimations obtaining a set of two parameters E_i and $v_i, \forall i = 1, 2$ that satisfy the load-deflection curve obtained experimentally. Then, with all parameters determined (E_i, σ_r, C_1) and experimental data, $C_{2(i)}$ is obtained with both Poisson's ratio found. So, parameters α, β are calculated as follows

$$\alpha = \frac{v_2 C_{2(1)}(1 - v_1) - v_1 C_{2(2)}(1 - v_2)}{v_2 - v_1}, \beta = \frac{C_{2(2)}(1 - v_2) - C_{2(1)}(1 - v_1)}{v_2 - v_1}. \tag{2}$$

Using α and β values, we can calculate any value of $C_2(v)$ with values of v known. With all parameters calculated until the step 7, the following error function can be mapped such as

$$e_{p(k)}(E, v) = \sum_{j=1}^{n} \left| \frac{P_{\exp(j)} - P(C_{2k}(v_k), E_k, w_{\exp(j)})}{P_{\exp(j)}} \right| / n \tag{3}$$

where subscript k means a set of parameters E_k and v_k determined for each load-deflection curve with n data. The result of Equation 3 is an error surface in which the minimum errors should be in the places where a set of E and v satisfy the experimental measurements. According to previous exploratory data analysis done in our study, it was found that a linear approximation can define the set of optimal solutions for E_k and v_k such as described in the step 8. Until this step, we found a set of parameters that approximate the analytical equation and the finite element model, this is due to that between both parameters elastic coupling exists. To compute a unique solution the following index is created

$$e_{c(k)}(E_k, v_k) = \sum_{i=1}^{m} \left| \frac{w^3_{\exp(j)}(x, y) - w^3_{FEM}(x, y)}{w^3_{\exp(j)}(x, y)} \right| / n, \tag{4}$$

Equation 4 was established to compare a 30% of the displacement field between the finite element models and the measured data. The minimum value $\min \left| e_{c(k)} \right|, \forall k = 1, 2 ..., p$ indicates that the elastic parameters (E^* and v^*) are the best approximations for the load-deflection curves obtained experimentally.

3 Results

For the application of proposed methodology, experimental tests were conducted for commercial silicon nitride films (Si_3N_4) with $2 \times 2mm$ of surface and $500nm$ of thickness. The tests were carried out in the apparatus described in Section 2.1, detailed information about it can be reviewed in [9].

Applying the procedures described until the step 6 (see Section 2.3), the following constants were determined; $\sigma_r = 166 \pm 2.5MPa, C_1 = 3.373, \alpha = 1.9690$ and $\beta = -0.4594$. Figure 3a shows the error function $e_p(E,v)$ established in Equation 2 which in turn was computed with the parameters anteriorly expressed in the domains $E \in (200,280)MPa$ and $v \in (0.2,0.4)$. It is observed that there is a region in which the elastic values minimize the function $e_p(E,v)$. As we proposed, these values listed in Table 1 are approximated by a linear relationship. The values indicate that all pairs satisfy load-deflection curve with good accuracy since these are mechanically coupled.

E [MPa]	v
220.7	0.322
230.4	0.286
232.4	0.278
235.4	0.267
240.0	0.250
245.1	0.230
250.2	0.211

Table 1: Elastic parameters that minimize $e_p(E,v)$.

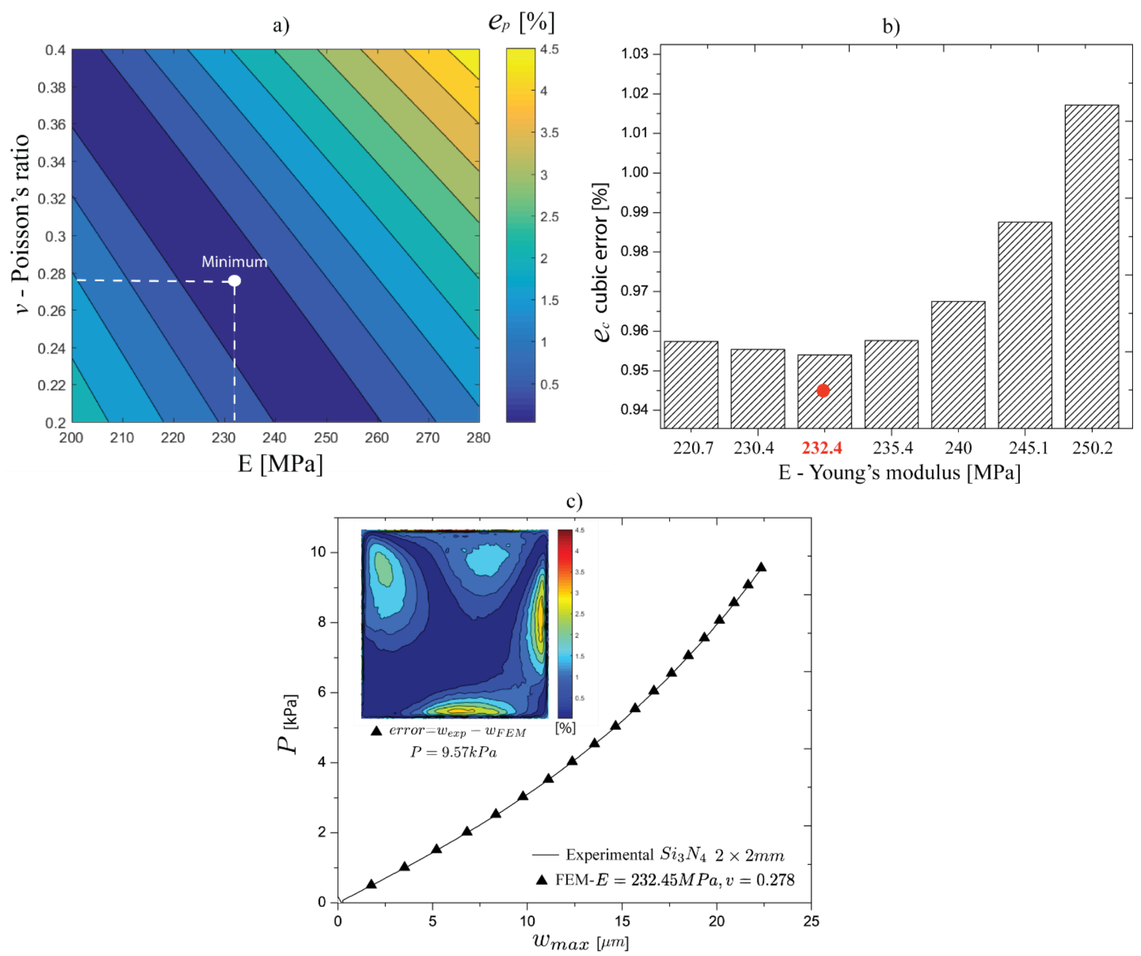

Figure 3: a. $e_p(E,v)$ error function. **b.** RMS cubic error scheme of the experimental setup. **c.** Load-deflection curve comparisons.

As described in step 9, values listed in Table 1 are used to perform finite element simulations to find an optimal solution inside the chosen values. These values represent a specific case of the experiments. Then, an optimal solution is found in the minimum of $e_c(E,v)$, for our test, it was determined as $E = 232.45 \pm 3MPa$ and $\nu = 0.278 \pm 0.01$ with five experiments analysed. The results are shown in Figure 3b.

In Figure 3c, it can be observed that the numerical solutions agree with the experimental data, computations were done to verify that calculated solution adjusts the measured data. Additionally, for the maximum state of pressure (9.57 *KPa*), the absolute error between finite element solution and experimental displacement field is evidenced. It is seen that results agree very well with the measurements since displacement errors are less than 1% in the majority of the bulge domain.

Conclusions

A numerical approach for identifying the elastic properties of thin films was described and applied, it based on bulge test analysis, classical analytical methods and finite element analysis. The main benefit lies in the determination of both properties Young's modulus and Poisson's ratio since in a traditional bulge test analysis only one of these can be determined. The proposed numerical procedure showed that comparing the deformed surfaces optimal elastic parameters can be found. Results showed that the estimated elastic properties agree with corresponding values reported by other methods in the literature.

Acknowledgement

This research has been supported by the Ministry of Education, Youth and Sports of the Czech Republic under the project m-IPMinfra (CZ.02.1.01/0.0/0.0/16_013/0001823) and the equipment and the base of research infrastructure IPMinfra were used during the research activities.

References

[1] Poelma, R. H., Sadeghian, H., Noijen, S. P. M., Zaal, J. J. M., & Zhang, G. Q. (2011). A numerical experimental approach for characterizing the elastic properties of thin films: application of nanocantilevers. *Journal of Micromechanics and Microengineering*, *21*(6), 065003.

[2] Pantano, M. F., Espinosa, H. D., & Pagnotta, L. (2012). Mechanical characterization of materials at small length scales. *Journal of Mechanical Science and technology*, *26*(2), 545-561.

[3] Mitchell, J. S., Zorman, C. A., Kicher, T., Roy, S., & Mehregany, M. (2003). Examination of bulge test for determining residual stress, Young's modulus, and Poisson's ratio of 3C-SiC thin films. Journal of Aerospace Engineering, 16(2), 46-54.

[4] Paul, O., & Gaspar J. (2007) Thin-film characterization using the bulge test, in Reliability of MEMS, O. Tabata and T. Tsuchiya, Eds. Weinheim, Germany: Wiley-VCH ch. 3, pp. 67–122.

[5] Xiang, Y., Chen, X., & Vlassak, J. J. (2005). Plane-strain bulge test for thin films. *Journal of materials research*, *20*(9), 2360-2370

[6] Tabata, O., Kawahata, K., Sugiyama, S., & Igarashi, I. (1989). Mechanical property measurements of thin films using load-deflection of composite rectangular membranes. *Sensors and actuators*, 20(1-2), 135-141.

[7] Vlassak, J. J., & Nix, W. D. (1992). A new bulge test technique for the determination of Young's modulus and Poisson's ratio of thin films. *Journal of Materials Research*, 7(12), 3242-3249.

[8] Maier-Schneider, D., Maibach, J., & Obermeier, E. (1995). A new analytical solution for the load-deflection of square membranes. Journal of microelectromechanical systems, 4(4), 238-241.

[9] Holzer, J.; Pikálek, T.; Buchta, Z.; Lazar, J.; Tinoco, H. A.; Chlupová, A; Kruml, T. (2017, May). Development of the bulge test equipment for measuring mechanical properties of thin films", In: *Proceedings of 26th International Conference on Metallurgy and Materials*. Brno, Czech Republic. pp. 1-6. Brno, Czech Republic.

[10] Pan, J. Y., Lin, P., Maseeh, F., & Senturia, S. D. (1990, June). Verification of FEM analysis of load-deflection methods for measuring mechanical properties of thin films. In: *Solid-State Sensor and Actuator Workshop, 1990. 4th Technical Digest., IEEE* (pp. 70-73).