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Numerical modeling approach for the assessment of elastic properties of bi-layer thin films measured by bulge test

Hector A. Tinoco[*a,b,c], Jakub Holzer [b], Tomáš Pikálek [d], Tomáš Fořt [d], Jaroslav Sobota [d], Zdeněk Buchta [d], Josef Lazar [d], Alice Chlupová [b] Tomáš Kruml [a,b], Pavel Hutař [a,b]

[a]*CEITEC-Central European Institute of Technology, Brno University of technology, Technická 10, 616 00 Brno, Czech Republic*

[b]*Institute of Physics of Materials, Czech Academy of Sciences, Žižkova 22, 616 62, Brno, Czech Republic*

[c]*Experimental and Computational Mechanics Laboratory, Universidad Autónoma de Manizales. Antigua Estación del Ferrocarril, Edificio Sacatín C.P. 170001. Manizales, Colombia*

[d]*Institute of Scientific Instrumen, Czech Academy of Sciences, Královopolská 147, 616 64, Brno, Czech Republic*

[*]Author to whom correspondence should be addressed. Phone: Tel + 420 532 290 338. E-mail: tinoco@ipm.cz

Abstract: The presented work shows a load-deflection bulge test method to characterize mechanical parameters of thin films by means of a numerical modelling approach. A methodology based on the combination of finite element analysis and classical analytical equations is presented and discussed. Finite element modeling was conducted for monolayer (Si_3N_4) and bi-layer (Si_3N_4/Al) membranes with the aim to determine a set of elastic parameters (Young's modulus and Poisson's ratio) which satisfied the load-deflection curves experimentally measured by the bulge test method. It is well known that in a traditional bulge test analysis only biaxial elastic modulus, i.e. combination of Young's modulus and Poisson's ratio, can be determined. It is due to the mechanical coupling that exists between the two elastic parameters. However, in this study both mentioned elastic constants can be determined independently through a proposed numerical procedure that includes error functions for minimizing the displacement with a specific set of optimal elastic parameters. Results shows that the estimated Young's modulus (Al 64 GPa and Si_3N4 236 GPa) agree with corresponding values determined by other methods in the literature of the studied thin films.

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

1. Introduction

Thin film manufacturing requires precise instrumentation to achieve the geometric and mechanical conditions necessary for each industrial application in which are microelectronics, nano-devices, coating applications, biomedical devices among others [1]. However, the evaluation of mechanical properties is important to predict the mechanical behaviour of thin films since these are subjected repeatedly to deformations by bending, folding, rolling, twisting, stretching, thermal cycling or compression. Therefore, to identify these properties with traditional material testing, it seems as a difficult task. Several experimental techniques have been developed to overcome challenges in the identification process of the properties; in which we can mention nanoindentation, diffraction-based techniques, Raman spectroscopy, deflection techniques among others [2-3]. The relative advantages and limitations of all the mentioned techniques have been discussed in the literature [3], which prefers the bulge test (deflection technique) because is easy to implement it. The bulge test consists in applying gas pressure on a free standing thin film and measuring the deflection over the bulged region [4-5]. This relation is called load-deflection curve and it is analysed by means of different methods that permits to determine the elastic parameters from the experimental data. For example, the fitting of analytical equations and finite element analysis [6-10] are the most used. This paper shows that elastic parameters (Young's modulus and Poisson's ratio) of a bi-layer Si_3N_4/Al film can be determined by combining finite element analysis with the classical method to identify properties.

2. Methods

2.1 Experimental setup for bulge tests in thin films

A bulge testing apparatus was constructed in-house with an adequate instrumentation and uses interferometric techniques to evaluate bi-layer (Si_3N_4/Al) membranes as shown in the scheme of Figure 1. Two important variables are measured in the bulge test, pressure and membrane deflection. The principle of the test is to apply a differential pressure on a free-standing membrane and to measure the membrane shape in form of displacement field for each pressure increment. The differential pressure is applied using an industrial grade piston that presses the air by computer-controlled syringe pump. Pressure is measured by the pressure transmitter (A) with maximal error of 60 Pa which is connected to a data acquisition system. The shape of the membrane is measured using an interferometric system (Twyman-Green interferometer) in which the light source is 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 of $\lambda/10$. The interferometric system uses a Nikon 50mm f/1.4 NIKKOR G lens to image the sample onto a camera that captures the interferograms that are used to determine the displacement field of the bulged film at the z position. In Figure 1a experimental setup is shown, and in Figure 1b, the interferometric system is illustrated with a reconstructed shape of a tested membrane Figure 1c. A complete description of the exposed bulge test apparatus is described in details in [9], basic working principles of bulge test can be found also in [2,4,6-7].

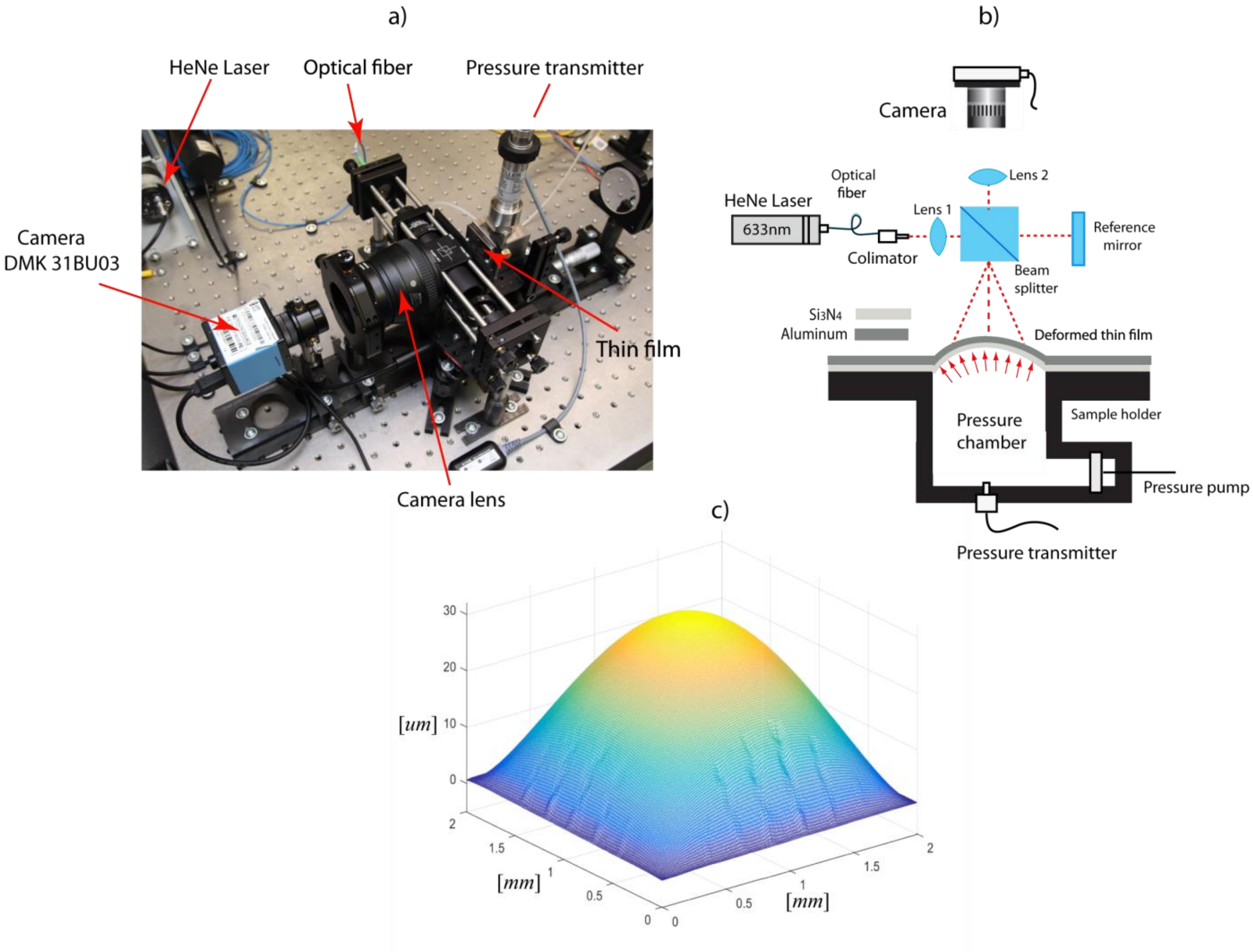

Figure 1. a) Experimental setup. b) Scheme of the experimental test. c) Reconstructed bulged bi-layer film of $2 \times 2\ mm^2$

2.2 Numerical strategy for determination of elastic parameters of bi-layer thin films

In this study, a numerical procedure is performed to determine Young's modulus and Poisson's ratio of bi-layer thin films combining finite element analysis with the following classical analytical solution [7]

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

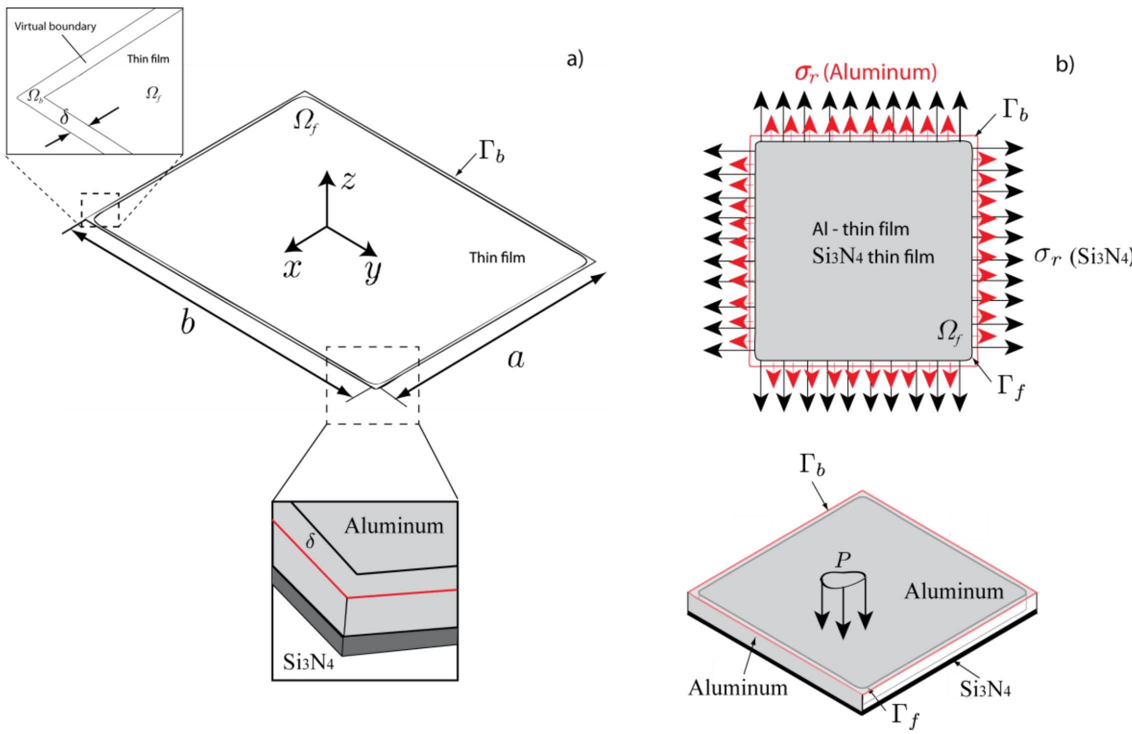

Figure 2. a) Segmented finite element model for bi-layer membranes (Si_3N_4/Al). b) Application of residual stress and load pressure in the finite element model.

where C_1 and $C_2(v)$ are constants that depend on geometric and material parameters, σ_r is the residual stress, t is the thickness, E is Young's modulus and v is Poisson's ratio. Equation 1 was initially proposed for monolayer membranes subjected to bulge test, but it can be extended to more than one layer [11-12]. A combination of numerical solutions and experimental data permits the determination of all elastic properties of bi-layer films in this study. In this process, we can highlight that finite element analysis is a powerful tool to simulate bulge testing technique with the aim to estimate the properties of thin films as demonstrated in different studies [2-7]. There are several challenges in the estimations of elastic properties until nowadays, as for example elastic coupling between Poisson's ratio and Young's modulus as it was evidenced by [10]. We propose estimation of the elastic properties in two stages; first stage, applying the procedure proposed by [10] in a monolayer. In a second stage, to determine the elastic properties of the studied membrane (knowing properties of the support membrane) by an iterative process with finite element analysis in bi-layer membranes.

For modelling of mono-layer and bilayer membranes by means of finite element analysis, a segmented geometry is considered to approximate real conditions in the simulation. The geometry is defined by the membrane Ω_f (real domain corresponding to experiments) and a virtual boundary δ (numerical boundary). Ω_f is composed by two layers which in our study are Si_3N_4 and Aluminium. With only the imposition of the residual stress σ_r on $\Gamma_{f(Si3N4)}$ is possible estimate the residual stress of the Aluminium by bending conditions with a proposed numerical strategy. Figure 2 shows the geometrical considerations for the finite element model and the application of the residual stresses, also details of this type of modelling can be reviewed in [10]. In this study, finite element solutions were obtained with ANSYS 16.1 on an ASUS ROG Strix GL553VE (Intel Core (TM) i7-7700HQ CPU @ 2.8 GHz, 16 GB RAM) notebook which was ran in Windows 10 environment in order to verify the proposed numerical procedures.

3. Results

Experimental bulge tests on Si_3N_4 (monolayer) and Si_3N_4/Al (bi-layer) of 2×2 *mm* (square) with thicknesses of 500 *nm* (Si_3N_4) and 1760 *nm* (Al) were carried out to be used as fitting database in the proposed methodology. These were tested with the experimental setup shown in Section 2.1. In Figure 3, the results (elastic properties) obtained for Si_3N_4 ($E = 236.22 \pm 2\ GPa, \nu = 0.264$) and Aluminum ($E = 64 \pm 2\ GPa, \nu = 0.3$) are shown. The comparison between load-deflection curves (experimental and numerical) is shown in Figure 3. It is seen that both results are well correlated since it agrees with finite element solutions. We can point out that the values determined for the elastic properties correspond to typical values reported in the literature for Si_3N_4 [7, 10,13] and Aluminum [14-15].

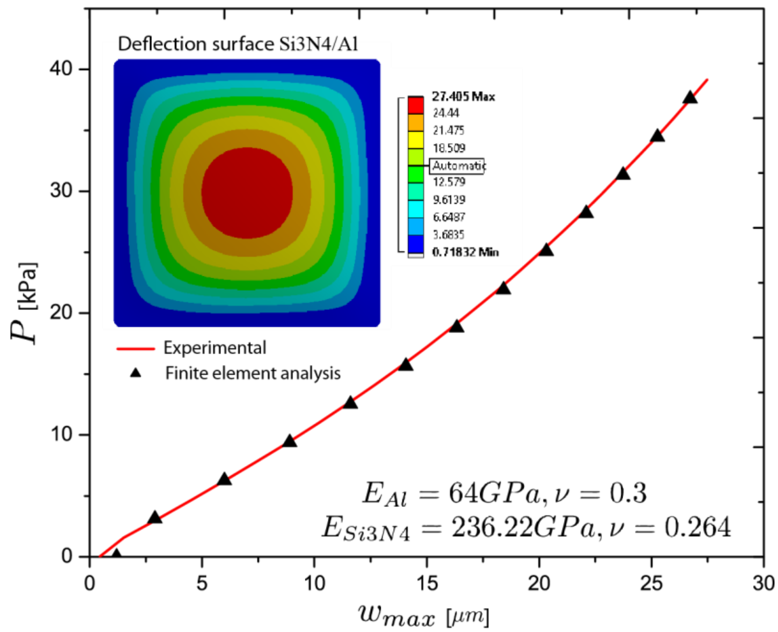

Figure 3. Comparison of pressure and maximum deflection curve

4. Conclusions

A numerical approach to determine elastic properties of bi-layer (Si_3N_4/Al) thin films was applied in this study. The analysis was based on bulge test (experimental technique), classical analytical method and finite element analysis, respectively. The main contribution is focused on the determination of both elastic properties (Young's modulus and Poisson's ratio) for bi-layer films since in a traditional bulge test analysis only a combination of these parameters can be determined. Results showed that the calculated elastic properties of both films agree with corresponding values reported in the literature.

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