Determination of the yield stress in Al thin film by applying bulge test

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Abstract. In this work, we have applied an improved method to determine the yield stress and residual stress in a freestanding thin aluminum film by analysing experimental data obtained by the bulge test. The Al thin film was deposited by a magnetron sputtering technique. The film was cyclically loaded with increasing maximum gas pressure. The method to determine the plasticity parameters is based on the load-deflection relation that presents a linear behavior in the elastic regime when it is scaled with the displacement parameter. The plastic deformation induces nonlinear effects that allow determining the elastic limit of the film. At that point, the gas pressure value that generates the elastoplastic transition is identified on the scaled curve. For a bulged square film, the curvatures are computed from an approximated spatial deflection equation to calculate the stresses within the proposed model. The analysis enables construction of biaxial stress-strain curve for the studied film and determination of the yield stress (132 MPa for the studied film). The second important development of the paper is the methodology which permits the characterization of the residual stress from pressure-stress relation without additional numerical computations.

1. Introduction

The mechanical characterization of thin films is essential for designing and developing microelectromechanical systems [1]. In thin films, the mechanical properties show a dependency on the manufacturing process (thermal growing, vapor deposition, etc.). Their exact determination leads to experimental challenges to be controlled on more dimensional scales (micro to nano) [2-3]. There are two types of prepared thin film specimens; those as-deposited on substrates, and others in freestanding conditions (removed substrate). Both present advantages and disadvantages depending on the experimental approach used to extract its intrinsic properties [4-6]. The bulge test is a well-known technique for studying several mechanical properties of thin films [7]. Generally, methods for determination of the residual stress, Young’s modulus, Poisson ratio, and fracture toughness, among
other properties, are reported for this technique [8–13]. Their implementation is considered a practical process since, compared with different techniques, there are less sophisticated procedures to be adopted. The experimental procedure is based on applying uniform pressure over one side of the freestanding film to cause its deflection out of its equilibrium position [14,15]. The relation between the pressure and the deflection is used for the characterization of mechanical properties under specific controlled conditions. For the stress computations, two types of specimen geometries are preferred: i) rectangular films (with aspect ratio \( b \geq 4a \)) are the most used since the curvatures are approximated by a cylindrical profile [16–18]; ii) circular ones treated within a spherical cap model [19–21]. The stresses at the location of maximum deflection depend on the pressure, and their local curvatures [22], which means that quasi-static (pressure) and kinematic (deflection field) parameters should be known. For square thin films, there are no reported analytical models to estimate the stresses and strains since the curvature computations have not been made available for the square bulged films; however, experimental evaluations have been reported for its determination [22,23]. This presents a limitation for the estimation of properties using the square membranes that depends directly on the stresses developed by the bulging effect as, for example, the yield stress. Studies of plastic properties of thin films are not common; however, some plasticity effects are known from the bulk materials, as the Bauschinger effect [24,25] or Portevin – Le Chatellier effect [26] were reported.

In this work, we used bulge testing combined with an analytical approximation to characterize the yield and the residual stresses from the experimental measurements. An Al thin film with a thickness of 1.74 μm was employed as the studied sample, which was subjected to cycling loading conditions. The presented methodology helps to found the elastic limit of the square thin films. The results are discussed with the aim to contribute to methods used for the elastoplastic characterizations of thin films.

2. Material and Methods

2.1. Determination of the elastic limit using the load-deflection curve obtained by bulge test

Bulge test is an experiment that uses uniform pressure over one side of a freestanding thin film deposited on a supporting material, as described in Figure 1a. After manufacturing, the film remains pre-stressed by residual stresses \( \sigma_y \). To represent the load-deflection behavior of an isotropic elastic thin film under the bulging experiment, Tabata et al. [27] proposed an analytical solution for the square films that relate \( w_0 \) (displacement at the center of the film) and \( P \) (pressure) as follows

\[
P = C_1 \frac{\sigma_y w_0}{a^2} + C_2(v) \frac{E w_0^3}{a^4},
\]

where \( C_1 \) and \( C_2(v) \) are constants that depend on the geometry and the material parameters; \( E \) and \( v \) are the elastic parameters (Young’s modulus and Poisson ratio); \( t \) and \( a \) are the thickness and the side length of the film. Several models and numerical estimations have been reported for both \( C_1 \) and \( C_2(v) \), as shown in Table I. Equation (1) is used for fitting the experimental data \( w_0 \) and \( P \), which are adjusted by the least-square fitting for computing \( \sigma_y \) and \( E \). This is possible if \( C_1 \) and \( C_2(v) \) are known. Tinoco et al. [28] demonstrated that there is a coupling between \( v \) and \( E \). It indicates that there is not a unique solution to satisfy \( C_1 \) and \( C_2(v) \) values. Equation (1) can be expressed in a scaled way, which shows its linear nature. By dividing both sides of Equation (1) by \( w_0 \), Equation (2) is obtained:

\[
Y = C_1 \frac{\sigma_y t}{a^2} + C_2(v) \frac{Et}{a^4} X,
\]

where \( Y = P/w_0 \) and \( X = w_0^2 \). Equation (2) is a linear expression that can be used to establish the limits of its applicability on real data, i.e. the elastic limit of the film [29,30]. According to Figure 2b,
the deviation from the linear part indicates the onset of plasticity, which induces nonlinearities in the graph.

**Table 1.** Values for $C_i$ and $C_j(v)$.

| $C_i$           | $C_j(v)$                                                                 | reference |
|-----------------|--------------------------------------------------------------------------|-----------|
| 3.044           | $\frac{\pi^6}{32(1+\nu)} \left[ \frac{5}{64} + \frac{(5-3v)^2}{9\pi^2(\nu-9) - 64(1+\nu)} \right]$ | [27]      |
| 3.41            | $1.981 - 0.585\nu$                                                      | [31]      |
| 3.393           | $\frac{1}{(0.8 - 0.062\nu^3)}$                                         | [32]      |
| 3.45            | $1.994 - 0.54\nu$                                                       | [33]      |
| 3.42            | $1.91(1 - 0.207\nu)$                                                   | [14]      |
| **By FEM**      | $(\alpha + \beta\nu)/(1 - \nu)$                                        | [28]      |

**Figure 1.** (a) Bulge test scheme. (b) Linear relation obtained from the load-deflection data.

A process to determine the deviations between $Y_l$ (linear) and $Y_m$ (measured) is proposed with the following procedure: 1) the first step is to construct a linear function $Y_l$ using the experimental data using the first part of the curve, 2) the second step is to compute the deviation of the linear elastic solution and the experimental data calculated as follows $\epsilon_r = (Y_m - Y_l) / Y_m$. It is now necessary to adopt a definition of the onset of plastic deformation; the $\epsilon_r$ value is used in the following way. The yield stress is reached when $\epsilon_r$ becomes larger than the threshold error $\phi: \epsilon_r \geq \phi$; at this moment, the values of parameters $P$ and $w_0$ are called limit pressure $P_l$ and limit displacement $w_0$. The parameter $\phi$ defines when the detectable plasticity effects are introduced in the bulged membrane. Their critical values were according to a numerical definition established posteriorly. It is important to remember that stress and strain are distributed inhomogeneously in the membrane. It means that the yielding starts in a certain area of the membrane and spreads along the membrane during ongoing loading. The next step is to identify the most loaded area of the membrane and calculate the local stress, which is considered the yield stress of the material. The following section describes this procedure.

2.2. *Estimation of the yield stress with the curvatures of Maier-Schneider et al.* [33]* model
One of the most important variables to measure in the bulge test is the displacement field of the membrane. It contains crucial information about the kinematic of the film at any pressure. In the literature, different approximations are reported to reproduce the shape of the bulged film. Timoshenko and Woinowsky-Krieger [34] presented a set of approximations based on series that posteriorly Maier-Schneider et al. [33] expanded in a model with two more terms to improve the proposed solution [34]. The modification improved the description of the bulged shape in the tested examples. Their general expression is valid for thin rectangular plates of a size $2a \times 2b$, where $a \leq b$. For the particular case of a square thin film $a = b$, it remains as

$$w(x, y) = \left( w_0 + w_1 \left( \frac{x^2 + y^2}{a^2} \right) + w_2 \frac{x^2y^2}{a^4} \right) \cos \left( \frac{\pi x}{2a} \right) \cos \left( \frac{\pi y}{2a} \right), \forall x, y \in (-a, a)$$

(3)

where the constants $w_1 = 0.401w_0$ and $w_2 = 1.161w_0$ depend on the maximum displacement $w_0$. Equation (3) is used to approximate the stress distributions of a bulged square thin film in our study. It is important to mention that a square film presents an equi-biaxial stress state, which is analogously applied for the curvatures, being $\kappa_{xx}(x, y) = \kappa_{yy}(x, y)$ which represents a symmetry condition in the kinematical development of the bulged film. Therefore, we calculate the curvature at the central point $\kappa_0$ from (4) as follows

$$\kappa_0(x, y, t) = -\frac{\pi^2}{4a^2} \cos \left( \frac{\pi x}{2a} \right) \cos \left( \frac{\pi y}{2a} \right) \left[ w_0 + w_1 \left( \frac{x^2 + y^2}{a^2} \right) + w_2 \frac{x^2y^2}{a^4} \right] + \left( \frac{2w_2y^2}{a^4} + \frac{2w_1}{a^2} \right) \cos \left( \frac{\pi x}{2a} \right) \cos \left( \frac{\pi y}{2a} \right) -$$

$$\frac{\pi}{a} \cos \left( \frac{\pi y}{2a} \right) \sin \left( \frac{\pi x}{2a} \right) \left( \frac{2w_2xy^2}{a^4} + \frac{2w_1x}{a^2} \right)$$

(4)

Equation (4) represents the curvatures produced by the deflection surface at the central point of a square thin film. In this location, stresses are developed by the pressure effects and the deflection geometry, which in equilibrium conditions are approximated as

$$\sigma_{ii} = \frac{P}{2t\kappa_0}$$

(5)

where $\kappa_0 = \frac{1}{\rho_0}$, being $\rho_0$ the curvature radius and $t$ the thickness of the membrane. Similar to the kinematic conditions, the equi-biaxial stress state at the central point is represented by $\sigma_{xx0} = \sigma_{yy0} = \sigma_0$. Equation (5) means a classical solution reviewed by different authors [18,35,36], which shows the dependency of the stresses with the curvatures generated by the bulged surface. In order to compute the stresses, Equation (4) is combined with Equation (5), determining that

$$\sigma_0 = \frac{4Pa^2}{2\left( \pi^2w_0 - 8w_1 \right)} \approx \frac{3}{10} \frac{Pa^2}{w_0 t}, \text{ if } \sigma_{\sigma_{ss0}} < \sigma_0$$

(6)

We assume Sander’s shell theory [36] to estimate the strains, which consider a thin curved shell as a bulged membrane. Therefore, strains in the mid-plane are calculated with the contributions of mechanical effects caused by the axial loading, bending, stretching, and the nonlinear effects produced by large deformations;
\[
\varepsilon_{\alpha}(x, y) = \frac{\partial u_\alpha}{\partial x} + \frac{1}{2A} \left( \frac{\partial w(x, y)}{\partial x} \right)^2 + \frac{1}{A} \frac{w(x, y)}{\rho(x, y)}, \tag{7}
\]

where \( \partial u_\alpha/\partial x = \varepsilon_\alpha = (1-\nu)\sigma_\alpha/E \) is the residual strain caused by \( \sigma_\alpha \). Combining Equation \( (7) \) and Equation \( (3) \), the following expression is determined

\[
\varepsilon_0 - \varepsilon_\alpha = \frac{5\pi^2}{30\sqrt{3}} \frac{w_0^2}{a^2} \tag{8}
\]

Equation \( (8) \) represents strains at the central point in an equi-biaxial strain state. If the elastic constants are not known, the residual strain could not be computed from known residual stress. However, Equation \( (8) \) shows that, in this case, it is possible to compute the difference \( \varepsilon_0 - \varepsilon_\alpha \). The analytical equations presented in section 2.1 are used to identify the elastic limit with the pressure-deflection data.

### 2.3. Experimental setup for bulge test and square Al thin film preparation

The experimental setup was custom-built at the Institute of Scientific Instruments of Czech Academy of Sciences (Figure 2). It comprises a Twyman-Green laser interferometer with a wide collimated beam, pressure transmitter, pressure pump, and digital camera. The interferometer equipment uses a fiber-coupled HeNe laser with a wavelength of 633 nm. The laser beam is split into a measuring beam that reflects off the specimen surface and a reference beam that reflects off a reference mirror (surface flatness of \( \lambda/10 \)). The measuring beam interferes with the reference beam at the interferometer output and forms interference fringes projected onto the sensor via camera lens (Nikon 50mm f/1.4 Nikkor G). The test specimen was prepared following the procedure by Vlassak and Nix. [32]. A layer of amorphous silicon nitride layer with stoichiometry close to \( \text{Si}_3\text{N}_4 \) and thickness of 525 nm was deposited by low-pressure chemical vapor deposition on Si monocrystalline wafer. A square window of 2 x 2 mm was opened in the Si wafer by wet anisotropic etching. The exact dimension of the window covered by the nitride membrane was 2.02 x 2.03 mm. An aluminium film with a thickness of 1.74 \( \mu \)m was deposited by magnetron sputtering on the nitride (deposition was done for 141 minutes at 500 W at magnetron in oscillating regime with argon pressure of 0.38 Pa). Finally, the \( \text{Si}_3\text{N}_4 \) layer was etched out, so only the freestanding Al membrane was subjected to the bulge experiments.

![Figure 2. Experimental setup for the bulge test.](image)

The specimen was glued to a stainless-steel plate with a hole in the centre and mounted to the chamber via four screws. A computer-controlled industrial-grade piston operated by a syringe pump
was used to increase and decrease the chamber pressure and on the membrane. The chamber pressure was monitored by a pressure transmitter with a precision of 60 Pa.

![Figure 3](image_url)

**Figure 3.** (a) Example of 3D experimental displacement field, (b) Displacement fields for different pressure states.

The camera sensor, syringe pump, and pressure transmitter were connected by the acquisition system to control and record it. By subsequent analysis of the interference patterns, it is possible to calculate the displacement of every pixel in the normal direction to the specimen surface in relation to the pressure change, as illustrated in Figure 3. The shape of the whole membrane can be reconstructed, and displacement $w(x, y)$ as pressure function can be plotted for each pixel, including the central point of the membrane.

3. Results and discussion

For the bulge test, an aluminum film with a thickness of 1.74 µm and size of 2×2 mm was cycled six times in loading and unloading conditions, respectively. Each loading cycle incremented the gas pressure progressively by 10 kPa, starting at a maximum pressure of 10 kPa in the first cycle, and finalizing of 60 kPa, for the last cycle. The schematics of the loading conditions are given in Figure 4a; the measured displacement of the central point versus gas pressure is in Figure 4b.

![Figure 4](image_url)

**Figure 4.** (a) The schematics of the membrane loading in six cycles; (b) Pressure – deflection curves as the central point.
Figure 5a shows the measured data correlating the pressure and stress values at the membrane centre $\sigma_{xx,0}$ for the first four measured loading cycles. The stresses were computed with Equation (6). For the gas pressure close to zero, an initial value of stress corresponds to the residual stress $\sigma_r$, as illustrated in the magnified section A-A. The residual stress is not changing for the first three cycles; these values were used for calculation of $\sigma_r$ in as-received Al film as $82.5 \pm 0.2$ MPa. It is also visible that residual stress is changing after subsequent cycling due to the plasticity effects. The value of $\sigma_r$ was compared with values computed using Equation (1) and constants $C_i$ listed in Table 2, following the procedure described in Tinoco et al. [28]. Table 2 demonstrates that $\sigma_r$ calculated according to Equation (6) proposed for calculating the equi-biaxial stresses at the central point in square thin films is in good agreement with the values calculated by classical model Equation (1). The difference between the value found in his study and calculated using Equation (1) ranges between 0.43% and 10% for the oldest approximation of $C_i$. According to the above results, we consider as an advantage that the residual stress can be determined without complicated calculations of $C_i$, as this study proposes. More importantly, the validity of Equation (6) is successfully verified.

Table 2. Residual stress computations with known $C_i$ values.

| $C_i$  | $\sigma_r$ [MPa] | Error [%] | reference |
|-------|------------------|-----------|-----------|
| 3.044 | 91.56            | 10.98     | [27]      |
| 3.41  | 81.73            | 0.93      | [31]      |
| 3.393 | 82.14            | 0.43      | [32]      |
| 3.45  | 80.79            | 2         | [33]      |
| 3.42  | 81.49            | 1.22      | [14]      |
| ---   | 82.5 ±0.2        | ---       | Present study |

According to the results described for Figure 5a, $\sigma_r$ value changed after the third loading cycle with maximum gas pressure of 30 kPa, indicating irreversible deformation of the membrane. For verifying if the plastic effects occurred in this loading cycle, Equation (2) was adopted. Experimental data $X[w_i]$ and $Y[P/w_i]$ were fitted by (1) linear elastic function ($Y_l$) and (2) smoothed by a cubic polynomial function ($Y_Nl$). Both functions $Y_{Nl}$ and $Y_{l}$ are plotted in Figure 5b.

![Figure 5](image_url)  
**Figure 5.** (a) Pressure-stress curve for different loading cycles. (b) Scaled load-deflection relations and relative error for the third loading cycle.

The relative error function $\epsilon_r$ was calculated to establish when the linear relation is no more followed by experimental data. Comparing the results, the following criterion was chosen to define the threshold
error; if the final error achieves a value higher than errors computed through the domain, the error limit is defined on the last error peak, as illustrated in Figure 5b. We can analyze that the first two in the loading cycles, the final errors do not achieve overpass the higher error on the domain. The third cycle satisfies the mentioned conditions, and we establish an error limit is 0.27%. This corresponds with $X = 1280 \mu m^2$ which is related to the pressure 27.6 kPa, and the stress calculated is 132.18 MPa. This value represents the yield stress determined for the equi-biaxial stress state at the central point.

Figure 6a shows stress-strain curves for the six loading cycles; the strains are relative measurements, excluding the residual strain calculated according to equation (8). It is noted that from the fourth cycle, the stress-strain curves start to shift down from the value of residual stress of the as-received film, and the difference of residual stress at zero strain between loading and unloading values augments. It indicates that when the plastic deformations are introduced, and the hysteresis increased in the stress-strain curve. This effect has been reported e.g. by [15]. It means that the residual stress diminishes with each cycle showing a stress relaxation effect. In general terms, we can observe that the plasticity effects after the third cycle influenced the mechanical behavior in each bulging stage that was detected with the presented methodology. The effects caused by the plastic deformation could be explained from a microstructural analysis since it is well known that the yield strength of thin films can be affected by more mechanisms operating in the microstructure as changes in the dislocation density or (sub) grain size.

(a) Stress and strain curve for different loading cycles. (b) Residual deformations after unloading cycles.

Figure 6b illustrates how the plastic effects are visualized on the measured displacement field after the film bulged. In this case, the last four loading cycles were considered for the analysis, from 30 kPa until 60 kPa. It is observed that the film remains deflected in a non-uniform way, which shows the higher residual displacements at edges. Progressively, the residual deflections increase their values from 0.8 \( \mu m \) to 3.6 \( \mu m \). Usually, in plates subjected to a transversal loading pressure, the stresses are higher in the boundaries when these are in clamped conditions. Yang et al. [6] discussed that the local curvatures induced restriction of the bending moments developed at the boundaries. As a consequence, stresses are higher at these locations.

4. Conclusions
This work described a methodology to determine some mechanical parameters of thin films using the bulge test. The biaxial stress-strain curve, yield stress at the central point of a square thin film, and residual stress and its evolution were determined with high precision. The procedures were applied for
an Al film, where load-deflection measurements were performed during the bulging process in conditions of cycling loading. The obtained results showed that plasticity effects were detected in the third cycle. Non-linear (plastic) behavior of the film started at the stress value of 132 MPa; this value is the yield stress of the film in a condition of biaxial tensile loading. Increasing plasticity was detected in the next applied loading cycles, showing a non-proportional shifting of the stress-strain curve in each loading cycle, which indicated that the plasticity effects were cumulative. Further, an analytical model was presented to determine the residual stress values without the necessity to apply Equation (1). The result showed good agreement with other values obtained by using classical methods.

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