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An improved method for point deflection measurements on rectangular membranes

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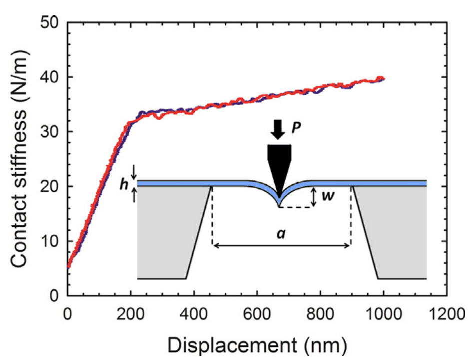
HIGHLIGHTS

- Residual stresses are measured from small deflection experiments on long rectangular membranes
- Continuous measurement of stiffness during nanoindentation improves reliability and repeatability of the experimental data
- The evaluation procedure is improved by adjusting the geometry factors to the elastic properties of the sample

GRAPHICAL ABSTRACT



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ABSTRACT

In this work, the recent theoretical advances for evaluating point deflection experiments are reviewed and further refined. An improved experimental approach based on measuring the contact stiffness during nanoindentation is implemented and applied to the evaluation of the residual stress in SiN_x and gold membranes. The accuracy of the point deflection experiments is assessed by evaluating the same set of samples with the bulge test reference technique and comparing the residual stress results. It is shown that the new experimental method greatly improves the reproducibility of the measurement and that the updated evaluation scheme leads to a more reliable residual stress value.

1. Introduction

Residual stresses are a detriment to the performance and lifetime of thin films and coatings. It is therefore essential to have a reliable method for their accurate measurement. X-ray diffraction [1] is most commonly used, but it is restricted to crystalline materials and relatively thick samples due to the need for a strong signal to noise ratio. Coatings as thick as a few micrometers can also be conveniently characterized by the combined Focused Ion Beam milling and Digital Image Correlation (FIB-DIC) approach [2–4]. Alternatively, residual stress can also be tested by the cantilever milling [5] and the wafer curvature [6] techniques. Measurements on submicron thin films, however, prove more difficult due to the lack of suitable techniques. The current method of choice is

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bulge testing [7–11], but this requires dedicated equipment not available everywhere. It would be more attractive to utilize a nanoindenter for mechanically deflecting the samples, since this equipment is already used for mechanical characterization and therefore more widely available. Such experiments were performed in the past on freestanding bridge structures [12–15], but these structures are tedious to fabricate. The aim of the current work is to present a reliable method for extracting the residual stress from point deflection experiments on long rectangular membranes, which can be fabricated by standard lithographic techniques. A direct comparison to bulge test measurements is provided in order to demonstrate the validity of the new nanoindentation-based method.

2. Point deflection theory

Point deflection tests are usually performed by loading a flat thin membrane with a nanoindenter tip, as schematically shown in Fig. 1. The evaluation of residual stresses from the nanoindentation data relies on a theoretical framework developed for the ideal case of a membrane being elastically deflected by the application of a load concentrated at its center point. A significant portion of point deflection knowledge can be traced back to the seminal work of Timoshenko *et al.* on plates and shells [16], which contributed three major aspects. First of all, Timoshenko derived the equation for plates free of residual stress subjected to small deflections [17]:

$$P = \frac{Eh^3}{12\alpha(1-\nu^2)a^2} w \tag{1}$$

E and ν are the Young's modulus and Poisson's ratio, respectively, while the other symbols are defined in Fig. 1. The parameter α is a factor that depends on the geometry, boundary conditions and load distribution of the plate. Values of α are known for common cases combining these three influences [16]. As this derivation is based on the classic Kirchhoff-Love theory, it is limited to pure bending, and only valid for deflections not exceeding the thickness of the plate. In the case of larger deflections, Timoshenko introduced a cubic term accounting for the stretching of the membrane, leading to the somewhat more complex equation:

$$P = C_1 \frac{Eh^3}{a^2} w + C_2 \frac{Eh}{a^2} w^3 \tag{2}$$

with the coefficients C_1 and C_2 known for a circular geometry, but not for the rectangular case. Timoshenko's third contribution was to propose a means for incorporating residual stress to account for its effect in the case of small deflections. Hong and Nix [18] explored this third aspect further and derived the exact solution for the small deflection of centrally loaded circular membranes with tensile residual stress:

$$P = \frac{4\pi}{3} \frac{Eh^3}{(1-\nu^2)a^2 gk} w \tag{3}$$

where k contains the information about the residual stress σ_0 and $g(k)$ is a function that asymptotically decreases from 1 (stress-free case) towards 0 with increasing residual stress. Although Hong's solution is exact, it did not prove very useful in practice due to the cumbersome fabrication of circular membrane geometries.

The case of rectangular membranes, which can be more conveniently fabricated by wet etching, was later developed by Poilane *et al.* [19]. Based on finite element simulations, they drew an analogy between the deflection behavior of circular and rectangular membranes. They suggested adapting Hong's solution (Eq. (3)) to the case of rectangular geometries by introducing a shape correction factor determined by finite element simulations. This factor was eventually formalized as β by Martins *et al.* [20]. Furthermore, they attempted to extend Hong's solution to large deflections by combining it with Timoshenko's constitutive equation for stress-free plates (Eq. (2)). The geometry-dependent coefficients were calculated for several geometries by Józwik *et al.* [21] and the final equation formalized by Martins *et al.* [20]:

$$P = \frac{Eh^3}{12\alpha(1-\nu^2)a^2 g(k)} w + C(\nu,k) \frac{Eh}{a^2} w^3 \tag{4}$$

where

$$k = \sqrt{\frac{12(1-\nu^2)}{\beta^2} \left(\frac{a}{h}\right)^2 \frac{\sigma_0}{E}} \tag{5}$$

$$g(k) = \frac{8}{k^2} \left[\frac{\left\{K_1(k) - \frac{1}{k}\right\}}{I_1(k)} \{I_0(k) - 1\} + K_0(k) + \ln\left(\frac{k}{2}\right) + \gamma \right] \tag{6}$$

$$C(\nu,k) = \frac{C_0}{1-\nu^2} \left(1 + \frac{\eta}{g(k)}\right) \tag{7}$$

I_0 and I_1 are the modified Bessel functions of the first kind of orders 0 and 1, respectively, and K_0 and K_1 the modified Bessel functions of the second kind of orders 0 and 1. γ is Euler's constant. The geometrical parameters provided by Martins *et al.* for the 4:1 rectangular geometry investigated in the current study are given in Table 1.

Eq. 4 is from the most current literature and therefore selected as the starting point for realizing an improved residual stress evaluation procedure. Two key tasks were identified for achieving this outcome, which will be addressed in the following paragraphs. Firstly, a finite element study is completed to verify the validity of the point deflection equations and refine knowledge about the geometry factors. Secondly, a direct comparison of the residual stress results with the reference bulge testing method is used to demonstrate the reliability of the improved experimental procedure against a known standard.

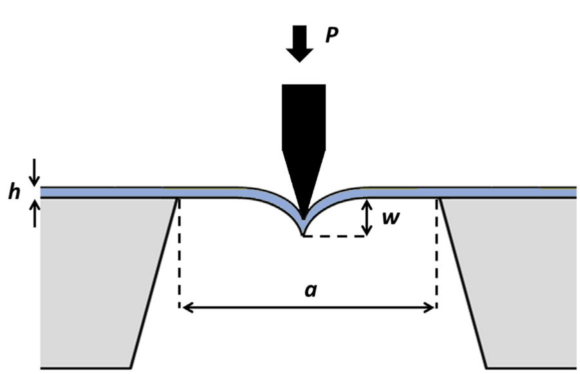

Fig. 1. Schematic cross section of a point deflection experiment on a freestanding membrane. P is the applied load, a is the freestanding span width, h is the membrane thickness and w is the deflection displacement at the center of the sample.

Table 1
Geometry factors provided by Martins *et al.* [20] for a 4:1 rectangular aspect ratio.

Geometry	α	β	C_0
Rectangular 4:1	$7.24 \cdot 10^{-3}$	1.58	not provided