ABSTRACT

In this paper we investigate, both experimentally and theoretically, the competition between different failure mechanisms (stretching, bending and curvature localization) in freestanding submicron thin films commonly used in micro-electromechanical systems. Micro-structures made of elastic-brittle materials such as ultrananocrystalline diamond, diamond-like carbon and silicon nitride, as well as elastic-plastic materials such as gold, aluminum, and copper, are tested by means of the membrane deflection experiment developed at Northwestern University. Evidence of competition between different failure modes has been found for the investigated elastic-brittle materials. The phenomenon is dependent on specimen size and shape. By contrast, in the case of elastic-plastic materials, failure due to stretching was found to be the dominant mechanism. An analytical model is proposed to rationalize the experimental data and to provide dimensionless parameters able to describe the competition between different failure mechanisms. These dimensionless parameters are particularly useful in the design of specimens employed in the MDE technique.

Keywords: Thin Films, MEMS materials, Fracture, Plasticity, Nanoscale Testing

1. INTRODUCTION

Thin films are customarily employed in microelectronic components and microelectro-mechanical systems (MEMS). Their properties frequently enable essential device functions. Therefore, accurate identification of these mechanical properties is instrumental to the development of innovative technologies. In the past ten years the advancement of mechanical testing of thin films has enabled extensive mechanical testing. The variety of techniques used in the tensile testing of thin films clearly illustrates its complexity. Recently a different test, known as membrane deflection experiment (MDE) was developed by Espinosa and coworkers. The technique involves the stretching of a free-standing thin film membrane in a fixed-fixed configuration with a double “dog-bone-like” shape. The geometry of the membrane is such that it contains tapered regions to eliminate boundary failure effects (Fig. 1). Several sizes of membrane specimens were designed on a single wafer (actual values of the dimensions are listed in Table 1).

![Fig. 1. Specimen with “dog bone-like” shape.](image1)

![Fig. 2. Schematic drawing of the MDE setup.](image2)
Table 1. Membrane dimensions for different sized specimens.

<table>
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<tr>
<th>Sample Geometry Type</th>
<th>A (µm)</th>
<th>B (µm)</th>
<th>C (µm)</th>
<th>D (µm)</th>
<th>E (µm)</th>
<th>F (µm)</th>
<th>G (µm)</th>
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<td>50</td>
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<td>100</td>
<td>100</td>
<td>200</td>
</tr>
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</table>

The membrane is attached at both ends and spans a micromachined window beneath. A nanoindenter applies a line-load at the center of the span to achieve deflection. Simultaneously, an interferometer focused on the bottom side of the membrane records the deflection (Fig. 2). The result is direct tension of a gauge region. Further details are given in Espinosa et al. [1]. The main advantage of the MDE test is its simplicity, the independent measurement of stress and strain, without recourse to mathematical assumptions or numerical interpretations of the experiments, and its accuracy and repeatability. The technique was employed to investigate the mechanical properties of brittle and ductile materials in the form of submicron free-standing thin films [1, 2]. Likewise, an extension of the MDE was employed to investigate fracture of MEMS materials [3] by introducing a sharp crack in the gauge region of the specimen, prior to its release, by means of microindentation.

A concern of the MDE test is the local bending of the specimen and its effect on material failure. Because of the specimen geometry, during the data reduction it is assumed that bending effects are localized and small, and that consequently they can be neglected. However, we have found that this assumption is not always valid. If the bending effect becomes important, it may be the case for very strong materials and certain specimen geometries (specimens with geometries C, D, and E), a competition between bending and stretching is ensued. When the deflection is dominated by bending, the specimen fails in the middle of the span region where the load is applied. On the contrary, when the deformation is dominated by pure tension, the specimen fails in the gauge region, as intended. In this work, we discuss the competition of bending and stretching in MDE specimens of various materials and geometries. An analytical analysis is presented to elucidate the experimental measurements and observations. The analysis confirms the existence of these competing failure mechanisms and provides dimensionless parameters that can be employed in the specimen design to avoid premature failure due to local or global bending.

2. EXPERIMENTAL OBSERVATIONS

Ultrananocrystalline diamond (UNCD), diamond like carbon (DLC), and Silicon Nitride (Si₃N₄) were tested as examples of elastic brittle materials. The failure mechanism of these three materials was found to be very similar. As an example we discuss in detail the case of UNCD. The material displays a linear stress-strain response up to fracture. Similar stress-strain behaviors were observed for DLC and Si₃N₄. The experimental results show that different failure modes occurred in different specimen geometries: stretching mode when failure occurred in the gauge region and bending mode when failure occurred in the middle of the span.

Fig. 3 shows: the strain-stress curve, recorded optical fringes, and the specimen failure mode. The figure also shows fractographic details obtained by means of scanning electron microscopy (SEM) of the fracture surfaces. Figs. 3a-d correspond to specimen type G, while Figs. 3e-h correspond to specimen type E. The strain-stress curves (Fig. 3e) are linear up to failure (note that the stresses and strains are calculated – see Section 3- in the gauge region, so their maximum values are not necessarily indicative of the strength of the material if failure occurs in bending mode). A Young’s modulus of 0.96 TPa and a fracture strength of 4.23 GPa (Fig. 3a) were observed for UNCD. The interferometric images reported in Figs. 3b,f show that the strain is uniform in the gauge region. The SEM images (Figs. 3c,g) show the existence of two failure modes. High magnification SEM imaging of the UNCD fracture surfaces reveals similarities with those observed in ceramic materials (Fig. 3i and 3k); namely, they exhibit a roughness comparable to the size of the grains clusters formed in the growth process [4]. Failure initiates from defects in the film and propagates between clusters.

From the stress-strain curve corresponding to the specimen exhibiting bending failure mode (Fig. 3e), the elastic modulus is again 0.95 TPa. The apparent strength is measured as 2.58 GPa. The interferometric optical fringes (Fig. 3i) shows that the local strain in the middle of the span, region where
the line load is applied, appears not uniform. Fracture occurs in this region and the fracture surface exhibits a kink. Similar features were observed in DLC and Si$_3$N$_4$ for the same type of specimen.

Fig. 3. Example of experimental results for elastic-brittle materials (UNCD) for stretching (left) and bending (right) failure modes: stress-strain curves (a and e), interferometric optical fringes (b and f), broken specimen (c and g) and crack surface (d and h).

Fig. 4. Example of experimental results for elastic-plastic materials (Copper) exhibiting stretching failure mode: stress-strain curves (a), optical fringes before failure (b) (note the non-homogenous spacing, showing a plastic localization in the thickness of the specimen), broken specimen (c) and failure zone (d).

The failure features observed in ductile materials are different from those previously discussed. Gold, aluminium and copper thin films with various geometries were tested under the same conditions. All films were deposited by e-beam evaporation. No specimen was found to fail in bending mode. In this case,
compatible film deflection is accomplished by development of plastic hinges at the fixed ends and in the middle of the span. Hence, the risk of failure due to bending is reduced. In Fig. 4, the same experimental results, previously discussed for the case of brittle materials are reported for the case of elastic-plastic materials (in particular for copper). The strain-stress curves of a 1 µm thick copper membrane with dimension F are given in Figs. 4a. A typical elastic-plastic behavior is observed. The Young’s modulus was measured to be 121 GPa; the yielding stress is about 250 MPa, whereas the failure stress is close to 465 MPa. The material exhibits a very high hardening rate and fails at relatively small strains. By examining the value of the stress at zero strain, the residual stress resulting from the microfabrication process is estimated to be around 50 MPa. The interferometric optical fringes clearly show failure in the gauge region (Fig. 4b). Fracture occurs in this region (Fig. 4c) indicating that tension dominates the failure competition. Fig. 4d is a high magnification SEM image of the failure region. Several features are apparent: the fracture exhibits a complex path with some clear changes in direction and the surface near the fracture is very smooth. The failure of the membrane can consequently be described as quasi-brittle in nature. Similar observations were made in Au specimens for thicknesses of 0.3 and 0.5 µm.

3. ANALYTICAL ANALYSIS

3.1 Stretching under large displacements

![Fig. 5. Three hinges model for MDE data reductions.](image)

MDE experiments are usually described by a three hinge model [1, 2] (Fig. 5). Considering the specimen of half-length \( l \), the strain due to the stretching \( \epsilon_s \) under large displacements (denoted by the angle \( \theta \), see Fig. 5) can be simply evaluated as:

\[
\epsilon_s = \frac{l - l \cos \theta}{l} = 1 - \cos \theta
\]

Eq. (1) represents the compatibility equation. In the linear elastic regime, the corresponding stress \( \sigma_s \) is:

\[
\sigma_s = E \epsilon_s = E (1 - \cos \theta)
\]

where \( E \) is the Young modulus. On the other hand, the tension \( T \) in the specimen under the applied load \( F \) is:

\[
T = \frac{F}{2 \sin \theta}
\]

Eq. (3) represents the equilibrium equation. The corresponding stress must be:

\[
\sigma_s = \frac{T}{A} = \frac{F}{2A \sin \theta}
\]

where \( A = WH \) is the film cross sectional areas, of width \( W \) and height \( h \) (the thickness of the film).

The stress-strain curve, according to Eq. (2), is expected to be linear only in the linear elastic regime. On the other hand, the force versus displacement can be obtained equating Eqs. (2) and (4):

\[
F = 2EA \left( 1 - \cos \left( \tan^{-1} \frac{c}{T} \right) \right) \sin \left( \tan^{-1} \frac{c}{T} \right)
\]

For not too large displacements ( \( \sin \theta = \tan \theta = \theta, \quad \cos \theta = 1 - \theta^2/2 \) ) it becomes:
As a consequence, the force-displacement curve is expected to be of a cubic form in the linear elastic regime. The experimental results clearly show this cubic form as an evidence of the imposed large displacements [1].

3.2. Competition between stretching and bending

In Section 3.1 we have assumed a mode shape corresponding to pure stretching. The complementary assumption corresponds to a mode shape of pure bending [5], i.e.:

$$w = -\left[2 \left(\frac{z}{l}\right)^3 + 3 \left(\frac{z}{l}\right)^2\right]c,$$  

(7)

where $0 \leq z \leq l$ is the axial co-ordinate starting from one clamp and $c$ is the central deflection. However, associated to this mode shape, stretching stresses and strains under large displacement arise. We study only half of the structure as a consequence of the symmetry.

To take into account the non linear effect of large displacements, we have to evaluate the energy stored in the beam not only due to bending but also due to stretching. The strain due to bending is:

$$\varepsilon_b = -\frac{1}{l} \frac{d^2w}{dz^2}$$  

(8)

where $y$ is a central co-ordinate (along a principal direction of the inertia tensor and with origin in the centroid of the cross-section), parallel to the direction of the applied force. Substituting Eq. (7) into (8) gives:

$$\varepsilon_b = 6 \left(\frac{z}{l} - 1\right) \frac{c}{l^2},$$  

(9)

The maximum value is reached at the external fibers where $y = \pm h/2$. The absolute maximum is:

$$\varepsilon_{b,\text{max}} = \frac{1}{l^2} \frac{c h}{l^2},$$  

(10)

and occurs at the coordinates $z = 0, l$ (i.e., at the fix ends and in the middle of the span).

In addition, the strain due to stretching is:

$$\varepsilon_s = \frac{1}{2} \frac{d w}{dc}$$  

(11)

Combining Eqs. (11) and (7) yields:

$$\varepsilon_s = 18 \left[2 \left(\frac{z}{l}\right)^3 - 3 \left(\frac{z}{l}\right)^2 + \left(\frac{z}{l}\right)^3\right] \frac{c}{l^2},$$  

(12)

Its maximum value will be reached at the point $z = l/2$ (i.e., in the gauge region):

$$\varepsilon_{s,\text{max}} = \frac{9}{8} \frac{c^2}{l^2},$$  

(13)

Note that the result of $\varepsilon_s = c^2/l^2$ is coherent with that of Eq. (1), under the assumption of moderate displacements.

The ratio between the stresses due to stretching and bending defines a dimensionless parameter describing the competition between stretching and bending:

$$\lambda_{sb} = \frac{\sigma_s}{\sigma_b} = \frac{\varepsilon_{s,\text{max}}}{\varepsilon_{b,\text{max}}} = \frac{3}{8} \frac{c}{h},$$  

(14)
From Eq. (13), $c_{u} = \lambda_{u} \sqrt{\lambda_{c}} \approx k_{1}$, so that:

$$\lambda_{u} = k_{2} \frac{f}{R_{1}} k_{1}$$

(15)

where $k_{1}$ is a constant that can be deduced from one experiment ($k_{2} = 126$). Note that $k_{2}$ cannot be determined theoretically (at least by this analysis), since it would vary considering different mode shapes for $u$ (e.g., as assumed in Eq. (7)). However, the validity of the result of Eq. (15) is general, since different mode shapes would result only in different value of $k_{1}$. If $\lambda_{u} \gg 1$, the predominant failure mechanisms is stretching, vice versa, for $\lambda_{u} \ll 1$, bending prevails. The analysis rationalizes the reason for which the different failure modes (stretching and bending) occur at, as predicted, different locations along the membrane length.

4. EXPERIMENTAL AND ANALYTICAL COMPARISON

In the membrane deflection experiment, the central vertical displacement $c$ and the applied force $F$ by the nanoindenter are measured. According to our analytical analysis, the stress is evaluated from Eq. (4) and the strain from Eq. (1). Note that this procedure does not assume a priori a particular constitutive law (e.g., linear elastic), so that experimentally stress-strain curves without material restriction (e.g., for linear elastic-plastic materials) can be obtained. As previously discussed, examples of computed stress-strain curves for brittle and plastic materials are reported in Figs. 3a and 4a, respectively. The optical fringes (recorded by the Interferometer Microscope) are used to define when the test begins, i.e., when the contact between nanoindenter and specimen is reached, to have an independent measurement of the displacements and to visualize the occurrence of strain localizations and/or fracture. The interference fringes for brittle materials exhibiting bending failure mode (Fig. 3) do not show any unusual pattern until abrupt failure occurs (Fig. 3e). On the other hand, for elastic-plastic materials, a strain localization in the middle of the gauge region (Fig. 4b), is manifested by a discontinuity in the fringes. Failure then occurs in this region (Fig. 4c). The failure localization is not observed in elastic-brittle materials even for stretching failure mode (Fig. 3c). Failure occurs catastrophically as expected.

Three different geometries for each of the three different investigated elastic-brittle (UNCD, DLC, Si$_3$N$_4$) and elastic-plastic materials (gold, aluminium and copper) are reported in Table 2. In this table each material and specimen geometry are specified and the corresponding dimensionless parameter is evaluated. The experimental observations for the two different failure modes are also emphasized (in the table “yes” means that we have observed the corresponding failure mode and “no” that we did not observe it). In agreement with our analysis the stretching was observed when $\lambda_{u} \gg 1$, whereas for $\lambda_{u} \ll 1$ bending prevailed. In some cases, for the same geometry and material, both stretching and bending failure modes were observed (in correspondence of $\lambda_{u} \approx 1$), resulting in completely different stress-strain curves. One of the tested Aluminium specimens is found to be close to a bending failure mode. Indeed the specimen exhibited a brittle failure, Espinosa et al., [6]. The larger values of this parameter for the majority of the elastic-plastic specimens represent the reason for which the bending failure mode was not observed.

5. CONCLUSIONS

In this paper we report experimental evidence of the competition between different failures mechanisms observed in freestanding submicron thin films tested by means of the membrane deflection experiment. The dominant failure mode was found to be stretching for each investigated size and shape only for elastic-plastic materials. By contrast, for elastic-brittle materials both failure modes were observed, as a function of the size and shape of the specimen. An analytical model is discussed to rationalize the se observations in terms of dimensionless parameters, which accurately characterizes the physics of the problem. In summary, the derived parameters should be used as a guide in the design of MDE specimens to achieve failure in pure tension. This optimized specimen design simplifies the experimental investigation by the MDE technique when brittle materials or ductile materials that may exhibit high yield stresses due to size scale effects —of increasing interest in MEMS component applications — as it is the case for diamond films in their various forms, shape memory alloys and composites.
Table 2. Competition between stretching and bending failure modes for the investigated elastic–brittle and elastic–plastic materials: the dimensionless competing parameter \( \tilde{k}_{cp} \approx \frac{L^2}{2S} \). L is the length of the gauge region (Fig. 1).

<table>
<thead>
<tr>
<th>Material</th>
<th>L [\mu m]</th>
<th>I [\mu m]</th>
<th>H [\mu m]</th>
<th>( \tilde{k}_{cp} )</th>
<th>Stretching</th>
<th>Bending</th>
<th>( \tilde{k}_{cp} )</th>
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REFERENCES