A Membrane Deflection Fracture Experiment to Investigate Fracture Toughness of Freestanding MEMS Materials

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ABSTRACT

This paper presents a novel Membrane Deflection Fracture Experiment (MDFE) to investigate the fracture toughness of MEMS and other advanced materials in thin film form. It involves the stretching of freestanding thin-film membranes, in a fixed-fixed configuration, containing pre-existing cracks. The fracture behavior of ultrananocrystalline diamond (UNCD), a material developed at Argonne National Laboratory, is investigated to illustrate the methodology. When the fracture initiates from sharp cracks, produced by indentation, the fracture toughness was found to be 4.7 MPa $m^{1/2}$. When the fracture initiates from blunt notches with radii about 100 nm, machined by focused ion beam (FIB), the mean value of the apparent fracture toughness was found to be 7.2 MPa $m^{1/2}$. Comparison of these two values, using the model proposed by Drory et al. [9], provides a correction factor of 2/3, which corresponds to a mean value of $\rho/2x=1/2$.

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INTRODUCTION

Significant research has been focused on investigating the mechanical properties of the structural materials for surface-micromachined MEMS devices. One such mechanical property which has been extensively studied is the fracture strength. However, for most practical uses of MEMS materials, an additional engineering parameter of importance is the fracture toughness, K_{IC}, which provides an assessment of the resistance a material possesses to crack growth from a pre-existing defect [1, 2]. K_{IC} depends only on microstructural factors and not on the specimen geometry, boundary conditions and loading. Consequently, the best way to compare structures of different geometries is on the basis of their respective stress intensity levels.

There have been a few recent reports on investigating the fracture toughness of polysilicon MEMS test specimens. Kahn et al. [3] and Ballarini et al. [4] used polysilicon fracture specimens integrated with on-chip fabricated electrostatic comb-drive actuators. The devices were tested using DC electrostatic actuation, while the displacements were measured by employing an optical microscopy with an accuracy of ~0.3 μm. The measured fracture toughness of polysilicon exhibited a mean value of 1.1 MPa $m^{1/2}$, which was found to be independent of polysilicon microstructure. Sharpe et al. [5] and Tsuchiya et al. [6] employed external piezoelectric load cells to fracture their notched specimens, and reported a critical stress intensity factor, K_{IC}, of 1.4 and 1.9 to 4.5 MPa $m^{1/2}$, respectively. These high values of K_{IC} are associated with finite radius (1.0 and 0.23 μm, respectively) of the notches and thus do not represent the true fracture toughness of the material. Chasiotis and Knauss [7, 8] performed tensile tests on perforated micro-specimens to investigate the fracture strength of polysilicon. Freestanding "dog bone-like" tensile specimens were loaded by means of an inchworm actuator until failure. A dedicated controller provided a measurement of the system displacement and the induced load was

measured by a miniature tension/compression load cell possessing an accuracy of 10^{-4} N and maximum load capacity of 0.5 N. The stress intensity, K_I, was obtained by this method using measured stress and deformation fields. However, limitations imposed by photolithography restricted the minimum radii of curvature, used in the perforated specimens, to one micron. As a result fracture toughness was not measured but rather the material strength at stress concentrations.

As various techniques were used to create the notches on the specimens results on K_{IC} varying from 1.1 to 4.5 MPa $m^{1/2}$ were reported by these researchers. This illustrates the need of a correction to take into account that a notch results in stress intensification rather than a singular field. Drory et al. [9] reported an analysis to obtain K_{IC} from blunt specimens. However, the fracture toughness identified with this procedure is only a lower bound estimate.

In this work a new membrane deflection fracture experiment (MDFE) [10] was used to investigate fracture toughness. As an example, the toughness of Ultrananocrystalline Diamond (UNCD) thin films is investigated. UNCD is a material developed at Argonne National Laboratory with unique properties particularly suitable to the development of novel MEMS/NEMS [11, 12]. The high stiffness (Young's modulus of 960 GPa [12]) and brittleness of UNCD make the measurement of its facture toughness particularly challenging. However, the MDFE here reported is capable of providing sufficient force and elongation to ensure crack propagation. Two types of specimens are tested and compared: blunt-notched specimens with crack tip radii of ~100 nm and cracked specimens. The theory by Drory et al. [9] is used to account for the effect of notch radius. The results are discussed in the context of MEMS applications.

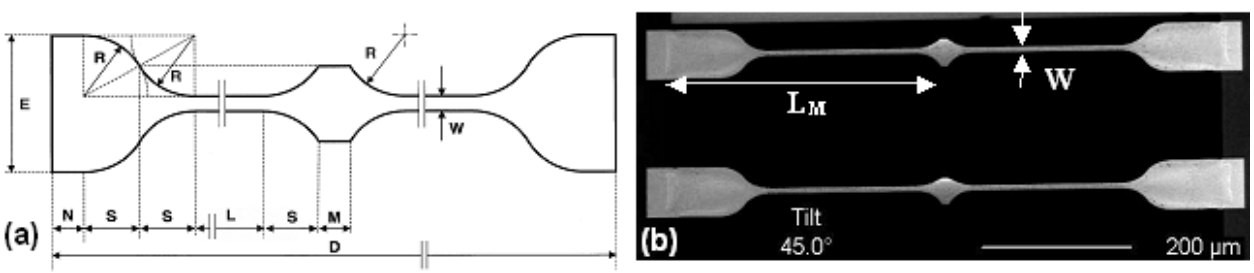

Fig. 1. (a) Schematic of membrane geometry indicating the different parameters used to define specimen dimensions. (b) Scanning electron microscopy (SEM) image of UNCD membranes showing characteristic dimensions. L_M is half the membrane span, and W is the membrane width.

EXPERIMENTAL METHODOLOGY

A. Specimen design and microfabrication

The specimen geometry utilized in this study resembles the typical dog-bone tensile specimen but with an area of additional width in the specimen center designed to prevent failure at the point of application of a line load (see Fig. 1.a) [10]. The suspended membranes are fixed to the wafer at both ends such that they span a bottom view window (Fig. 1.b). In the areas where the membrane is attached to the wafer and in the central area the width is varied in such a fashion to minimize boundary-bending effects. These effects are also minimized through large specimen gauge lengths. Thus, a load applied in the center of the span results in direct stretching of the

membrane in the two areas of constant width in the same manner as in a direct tension test. In this study, membranes with nominal dimensions of length L_M=350μm, width W=20μm, and thickness, t=0.8μm, were tested (see Fig. 1.b).

The film was grown directly onto a Si substrate using a microwave plasma chemical vapor deposition technique based on a novel CH_4/Ar chemistry [13]. The specimens were microfabricated using standard processes [12].

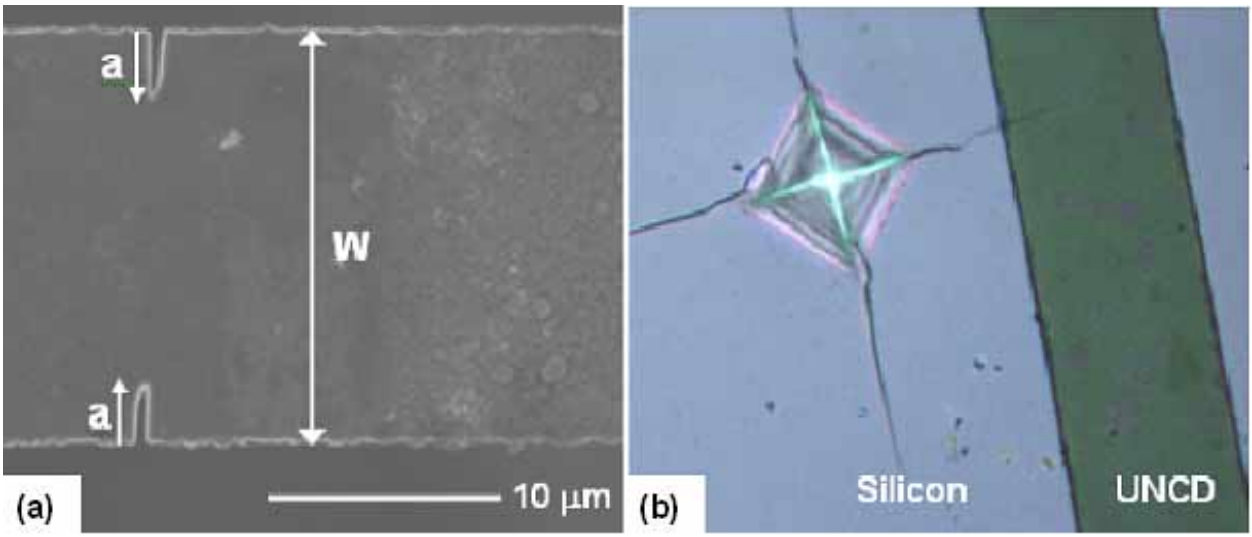

Fig. 2. (a) SEM micrograph of a pre-notched specimen milled by FIB with the two-symmetric-edge configuration. (b) Optical image showing the sharp crack induced from an indent on the silicon substrate before the specimen was released.

Two techniques were used to produce the pre-existing defects resulting in two types of specimens. The first one used focused ion beam (FIB) micro milling with a beam current density of 15 A/cm^2 and a minimum beam diameter of 10 nm. Two notches with the same length were milled simultaneously. A symmetric-edge cracks configuration was obtained. The geometry of the specimen is shown in Fig. 2.a. Due to the error and the vibration of the ion beam blunt notches with tip radii of ~100 nm were produced. In this configuration, an apparent fracture toughness can be computed from the following equations [14]:

$$K'_{IC} = \sigma_f \sqrt{\pi a}\, f(\frac{a}{W}) \tag{1}$$

$$f(\frac{a}{W}) = 1.12 + 0.43(\frac{a}{W}) - 4.78(\frac{a}{W})^2 + 15.44(\frac{a}{W})^3 \tag{2}$$

where σ_f is the failure stress, a is the length of the cracks and W is the width of the gauge region as shown in Fig. 2.a.

The second technique achieved an atomically sharp crack by placing a Vickers indent (with a 200g load) near the specimen prior to its release by KOH etching. Although the indent was placed on the silicon substrate (see Fig. 2.b) the radial cracks initiated at the corners propagated into the UNCD specimens. The length of the crack was measured using high resolution scanning

electron microscope (SEM). An edge-crack model was used to compute K_I for the second type of specimen. The function $f(\frac{a}{W})$ is given by:

$$f(\frac{a}{W}) = 1.12 - 0.23(\frac{a}{W}) + 10.55(\frac{a}{W})^2 - 21.72(\frac{a}{W})^3 \quad (3)$$

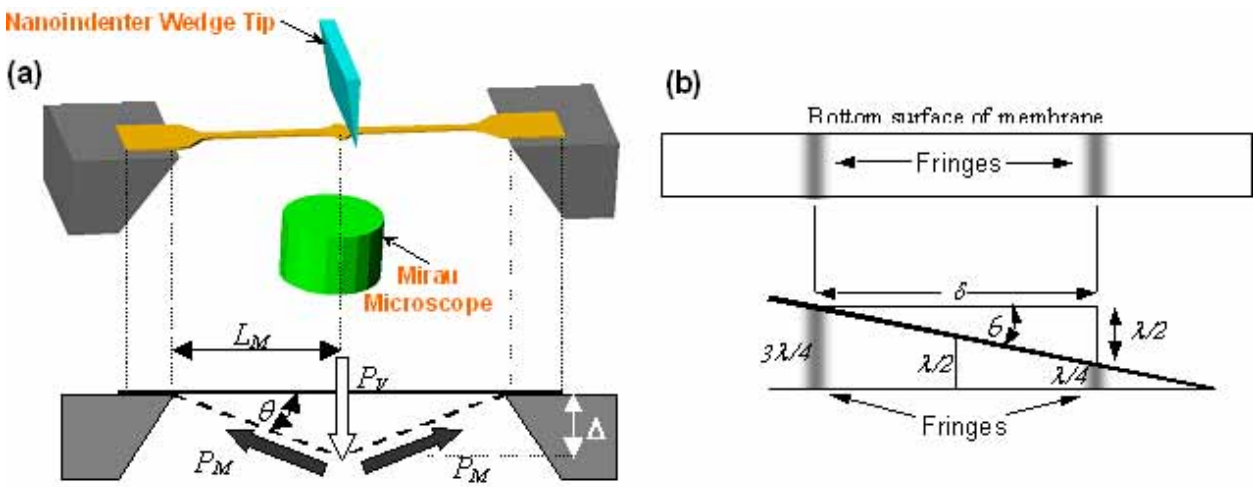

Fig. 3. (a) Schematic drawing of the MDFE setup. Parameters are defined in the text. (b) Schematic representation showing the relationship between distance between fringes (δ) and vertical displacement. The distance between fringes is taken at the central points of the dark bands.

B. Methodology

The Membrane Deflection Fracture Experiment (MDFE) was used to achieve direct tensile stressing of the specimen until failure [10]. The procedure involves applying a line-load, with a nanoindenter, at the center of the spanning membrane. Simultaneously, an interferometer focused on the bottom side of the membrane records the deflection. The result is direct tension of the two gauge regions with load and deflection measured independently. A schematic of the membrane deflection experimental set-up is shown in Fig. 3.a. It consists of a nanoindenter, to apply load to the center of the membrane from the top, and a Mirau microscope interferometer positioned directly below the specimen to independently measure deflection through the microfabricated die window.

The data directly obtained from the MDFE must then be reduced to arrive at a stress-strain signature for the membrane. The load in the plane of the membrane is found as a component of the vertical nanoindenter load by the following equations:

$$\tan\theta = \frac{\Delta}{L_M} \text{ and } P_M = \frac{P_V}{2\sin\theta} \quad (4)$$

where (from Fig. 3.a) θ is the angle of deflection, Δ is the displacement, L_M is the membrane half-length, P_M is the load in the plane of the membrane, and P_V is the load measured by the nanoindenter. Once P_M is obtained the nominal stress, $\sigma(t)$, can be computed from:

$$\sigma(t) = \frac{P_M}{A} \tag{5}$$

where A is the cross-sectional area of the membrane in the gauge region.

As the membrane is deflected by the nanoindenter, the interferometer, which works based on the Michelson Interferometer principle, records the membrane deflection by resolving surface fringes. A fringe will occur at each $\lambda/2$ change in vertical height of the membrane. The relationship corresponding to the distance between fringes, δ, and vertical displacement is shown in Fig. 3.b. Assuming that the membrane is deforming uniformly along its gage length, the relative deflection between two points can be calculated, independently of the nanoindenter measurements, by counting the total number of fringes and multiplying by $\lambda/2$. Normally part of the membrane is out of the focal plane and thus all fringes cannot be counted. We find the average distance between a number of fringes that are in the focal plane and then compute the angle θ. The average fringe distance, within the specimen gage region, is then obtained as $\delta=\lambda/(2\tan\theta)$. From this information an overall strain, $\varepsilon(t)$, for the membrane can be computed from the following relation, viz.,

$$\varepsilon(t) = \frac{\sqrt{\delta^2 + (\lambda/2)^2}}{\delta} - 1 \tag{6}$$

RESULTS AND DISCUSSION

Fifteen specimens with the FIB-machined notches were tested under the same condition except that the crack length, a, was varied from 1 to 5 microns. The ratio of the crack length and specimen width, a/W, is in the range of 0.05-0.25. The dimensions a and W were measured using scanning electron microscopy (SEM). The fracture toughness of the blunt notched specimens was then computed using equations (1) and (2).

Values of $K'_{IC} = 7.2 \pm 0.5$ MPa m$^{1/2}$ were obtained (Table I). From Table I it is clear that the apparent fracture toughness is independent of the crack length. This suggests that only the region of the material immediately in front of the crack like defect affects the fracture toughness. It is expected that the crack propagation speed would be very high because UNCD has a large elastic modulus and a low density.

It is important to note that in the computation of the K'_{IC} values, the notch was replaced by an apparent crack. Hence, corrections must be made because the effect of the blunt notch is to reduce the stress intensity relative to that ahead of a sharp crack. If this effect is not considered, resulting fracture toughness values become erroneously high.

From the above discussion the deviation from the sharp crack assumption of equation (1) needs to be corrected. For a blunt tip the asymptotic stress field is given by [15]:

$$\sigma_y(x) = \frac{K}{\sqrt{2\pi x}}\left(1 + \frac{\rho}{2x}\right) \tag{7}$$

Table I. Fracture Toughness measurement of 15 UNCD specimens.

Sample Number	a (μm)	σ_f (GPa)	K'_{IC} (MPa m$^{1/2}$)	K_{IC} with blunt notch correction (MPa m$^{1/2}$)
1	1.0	3.6	7.3	4.9
2	1.7	2.9	7.5	5.0
3	1.7	2.6	6.9	4.6
4	2.0	2.4	6.8	4.5
5	2.1	2.5	7.4	4.9
6	2.2	2.4	7.1	4.7
7	2.3	2.3	7.0	4.7
8	2.4	2.2	7.0	4.7
9	2.7	2.2	7.2	4.8
10	3.5	1.8	6.9	4.6
11	3.5	1.9	7.3	4.9
12	3.7	1.9	7.7	5.2
13	4.0	1.7	7.0	4.6
14	4.1	1.8	7.4	4.9
15	4.9	1.6	7.3	4.9
Mean values	---	---	7.2	4.7

where the origin of the reference system is in the middle between the tip and the center of the circular blunt notch, so that $x > \rho/2$. Comparing this stress field with the corresponding one for a sharp crack ($\rho \to 0$), Drory et al. [9], proposed the following correction:

$$K_{IC} = \frac{K'_{IC}}{1 + \frac{\rho}{2x}} \tag{8}$$

in which K'_{IC} is the measured fracture toughness from a blunt notch, ρ is the notch root radius, and x is a radial distance. Since the value of $\rho/2x$ is not determined, Drory et al. [9] assume $\rho/2x$ to be 1 providing a lower bound for K_{IC}. Consequently, they propose a 50% reduction on the measured fracture toughness to take into account the presence of the blunt tip [9]. By measuring the toughness in a specimen containing an indentation induced crack an assessment of this correction can be made. As shown in Fig. 3.d, cracks produced by indentation can be assumed atomically sharp, therefore, Eqs. (1) and (3) can be employed to compute the fracture toughness of the material. Several experiments performed on specimens containing these cracks, resulted in a toughness K_{IC}= 4.7 MPa.m$^{1/2}$. Therefore, for an average K'_{IC} of 7.2 MPa.m$^{1/2}$, we find that $\rho/2x$ should be 0.52, which coincides with the mean value of $\rho/2x$, which is 1/2. Consequently we propose to employ a correction factor in Eq. (8) of 2/3. The corrected values are shown in Table I.

CONCLUSION

A new methodology for the fracture toughness measurement of thin films and MEMS materials was presented. The membrane deflection fracture experiment (MDFE) is a simple and robust method to investigate fracture toughness of MEMS materials in mode I. The technique

can be used to interrogate both cracks obtained by indentation and notches microfabricated by FIB or other techniques. In this investigation we obtained notches about 100 nm in radius. It is possible that the sharpness of the notch can be improved by using a more precise focused ion beam system or other milling parameters. Fracture toughness of UNCD thin films was consistently measured. A correction to take into account the presence of the blunt tip is also proposed. Results indicate that the fracture toughness of UNCD is independent of the crack length as expected from fracture mechanics theory. The toughness of UNCD films established in this work indicates that the material is ideal for the development of novel MEMS/NEMS devices. Future work will focus on fracture test on different materials to investigate the relationship between toughness and microstructures.

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