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A methodology for determining mechanical properties of freestanding thin films and MEMS materials

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Abstract

We have developed a novel chip-level membrane deflection experiment particularly suited for the investigation of sub-micron thin films and microelectro-mechanical systems. The experiment consists of loading a fixed-fixed membrane with a line load applied at the middle of the span using a nanoindenter. A Mirau microscope interferometer is positioned below the membrane to observe its response in real time. This is accomplished through a micromachined wafer containing a window that exposes the bottom surface of the specimen. A combined atomic force microscope/nanoindenter incorporates the interferometer to allow continuous monitoring of the membrane deflection during both loading and unloading. As the nanoindenter engages and deflects the sample downward, fringes are formed and acquired by means of a CCD camera. Digital monochromatic images are obtained and stored at periodic intervals of time to map the strain field. Stresses and strains are computed independently without recourse to mathematical assumptions or numerical calibrations. Additionally, no restrictions on the material behavior are imposed in the interpretation of the data. In fact, inelastic mechanisms including strain gradient plasticity, piezo and shape memory effects can be characterized by this technique.

The test methodology, data acquisition and reduction are illustrated by investigating the response of 1-µm thick gold membranes. A Young's modulus of 53 GPa, a yield stress of 55 MPa and a residual stress of 12 MPa are consistently measured. The post-yield behavior leading to fracture exhibits typical statistical variations associated to plasticity and microcrack initiation. © 2002 Elsevier Science Ltd. All rights reserved.

Keywords: Thin films; MEMS materials; Micro-tensile test; Mechanical properties

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1. Introduction

Thin films are customarily employed in microelectronic components and microelectromechanical systems (MEMS) devices. Their properties frequently allow essential device functions and therefore accurate identification of these properties is key to the development of new technologies. In some applications, the demands placed on thin films can sometimes subject them to various mechanical conditions leading to fracture, plasticity, friction and wear, creep, fatigue, etc. Unfortunately, most of our knowledge in these areas is based on bulk material behavior, which many times fails to describe material response in this size regime. This is the case due primarily to surface and interface effects, limited number of grains in a given volume, and the role played by the manufacturing process. This last feature is very important in view that specimen surfaces are the result of the process employed to remove material. For instance, dry and wet etching lead to differences in surface roughness. Likewise, different chemicals utilized as etchings as well as process parameters, such as temperature and time, contribute to produce surfaces with well-defined features and defects, e.g., striations. Many researchers are currently investigating the mechanical response of thin films, e.g., Yuan and Sharpe (1997), Vlassak and Nix (1992), Chasiotis and Knauss (1998), Drory and Hutchinson (1995), Evans et al. (1997), and Huang and Spaepen (1996). Frequently, each particular investigation involving MEMS tends to be device dependent and introduces new fundamental questions. Progress in this field has leaned towards providing more specific technological solutions rather than generating a basic understanding of mechanical behavior.

Techniques to study MEMS materials' response to mechanical loading are diverse and can be classified as static or dynamic. Although both will yield the materials' mechanical properties, they accomplish it in completely different manners. Within the static group are nanoindentation (in standard DC mode), (Nix, 1989, Oliver and Pharr, 1992), micro-tensile (Yuan and Sharpe, 1997) bending (Sharpe, 1995, 1996; Zeng and Sharpe, 1996) and bulge tests (Small and Nix, 1992; Vlassak and Nix, 1992; Small et al., 1994). Nanoindentation, when the continuous stiffness measurement feature is used, resonance and fatigue methods (Kiesewetter et al., 1992; Osterberg and Senturia, 1997; Manceau et al., 1996) belong to the dynamic group.

Conventional understanding of material yielding and fracture does not apply at this scale because of the increased role that interface-driven processes play. Thus, there is a need to establish novel testing methodologies that measure stress and strain directly and independently on a variety of specimen geometries. The equivalent of a tensile test customary performed on bulk samples is desirable in this regard. Loads and strains are measured directly and independently, and no mathematical assumptions are needed to identify quantities describing the material response. Techniques that use a special fixture to load small tensile samples have been developed (Sharpe, 1995, 1996; Zeng and Sharpe, 1996; Chasiotis and Knauss, 1998). These techniques employ a dog-bone type specimen that is fixed at one end and freestanding at the other end. A probe is attached to the freestanding end to elongate/load the specimen. Sophisticated procedures for attaching the probes, by employing electrostatic forces or UV curing glues, were developed by Sharpe at Johns Hopkins University and W. Knauss at Caltech. However,

stress-strain curves cannot be uniquely determined when the various techniques are compared (Sharpe et al., 1998). This is due in part to microfabrication steps, such as chemical etching, which affect the specimens and tests' outcome in different manners. Note that specimens employed in different testing techniques have not only differences in geometry but also in microfabrication steps.

An ideal architecture to achieve a direct tensile testing scheme involves a freestanding membrane fixed at both ends. A line load applied at the middle of the span produces a uniform stretch on the two halves of the thin membrane. We have demonstrated this testing scheme by the investigation of radio frequency (RF) MEMS switches, produced by Raytheon Systems Co. (Fischer, 1999; Espinosa et al., 2001a–c). In this method we made use of a nanoindenter to apply a line load at the center of the membrane. Pushing the membrane down tests the specimen structural response and provides information on its elastic behavior and residual stress state. In this manner, simple tension of the membrane is achieved except for boundary bending effects.

A critical concern in this membrane deflection experiment (MDE) was accounting for the thermal drift and spring constant of the nanoindenter column. Since the column dimension is orders of magnitude larger than the membrane deflection, minute changes in temperature, a fraction of a degree C, can significantly affect displacement measurements. To account for these two factors, we made indents on either post supporting the membrane. Corrections for thermal drift and spring constant were then calculated from the approach segment data before contact with the posts. Other important information is also gained from these indents such as; device tilt, height of the membrane at contact, and middle position in the plane of the film. The load-displacement data can then be adjusted accordingly. We use a similar testing methodology here to examine elasticity, plasticity and fracture of thin films although the specimen geometry and wafer are modified to achieve homogeneous deformations.

The paper is organized as follows. The specimen design and microfabrication procedure are first discussed. Details on testing methodology and setup are then given with particular attention to features that affect the measurements accuracy. The algorithm for data reduction, accounting for the optical path of the interferometer light and membrane geometry, is presented and followed by discussion of experimentally obtained stress-strain curves in gold films. Test repeatability and size effects on elasticity, plasticity and fracture are addressed to illustrate the potential of the developed chip-level experiment.

2. Membrane deflection experiment (MDE)

2.1. Specimen design

The specimen geometry utilized in this study resembles the typical dog-bone tensile specimen but with an area of additional width in the center designed as the contact area where the line load is applied, Fig. 1. This measure is taken to minimize stress concentrations where the loading device contacts the membrane. The suspended membranes are fixed to the wafer at either end such that they span the bottom view window.



Fig. 1. Optical image of three Au membranes showing characteristic dimensions. L_M is half the membrane span, and W is the membrane width.

In the areas where the membrane is attached to the wafer and in the central contact 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 areas of thin constant width in the same manner as in a direct tension test.

Several types and sizes of membrane specimens were designed on a single wafer. Fig. 2 is a schematic drawing indicating particulars of the membrane dimensions. Actual values are listed in Table 1. The different types and sizes of membranes are represented in individual windows of each die, 68 dies per 4 in wafer, where membranes are placed 5 per window, Fig. 3. In this study we will report on only two different sized tensile specimens as a proof of concept for this methodology, tensile specimens "d" and "e". These two specimens possess identical shape, but at different length scales.

2.2. Micro fabrication of specimens

The suspended membrane specimens were fabricated on (100) Si wafers with double sided polishing. Fig. 4 is a schematic drawing summarizing the microfabrication steps. A layer of Si₃N₄ was deposited in both sides of the wafer to act as etch stops to aid in defining the bottom view windows and to protect the membranes during wet etching of Si. Windows were then dry etched into the Si₃N₄ on the bottom side.



Fig. 2. Schematic of membrane geometry indicating the different parameters used to define specimen dimensions.

Dimensions (µm)		Sample type							
		Tensile specimens					Fracture specimens		
		A	В	С	D	Е	F	G	Н
	± 2	514	362	286	1425	818	514	818	1425
Ε	± 0.5	45	22.5	11.25	180	90	45	90	180
L	± 0.5	100	50	25	400	200	100	200	400
W	± 0.1	5	2.5	1.25	20	10	10	20	40
M	± 0.5	10	10	10	10	10	10	10	10
N	± 1	100	100	100	100	100	100	100	100
R	± 0.5	20	10	5	80	40	20	40	80
S	± 0.5	17.32	8.66	4.33	69.28	34.64	17.32	34.64	69.28
Н	± 0.1	_	_	_	_	_	1	2	4.5
	± 0.1	_	_	_	_	_	1	1	1
$L_{\rm W}$	± 2	2078.5	2078.5	2078.5	2078.5	2078.5	2078.5	2078.5	2078.5
WW	± 2	1112.7	960.8	884.8	2024.5	1416.7	1112.7	1416.7	2024.5
Limit(-)	_	333.9	182	106	1245.7	637.8	333.9	637.8	1245.7
Limit(+)	—	404.7	252.8	176.8	1316.5	708.6	404.7	708.6	1316.5

Table 1 Membrane dimensions for different sized specimens

Patterning of the membranes on the topside of the wafer was accomplished by lift-off. A negative photoresist was exposed and developed followed by e-beam evaporation of a few nanometers of Ti, to aid adhesion, and then Au to a specified thickness (0.25 $-1 \mu m$). A Teflon chuck was used to protect the top surface of the wafer during wet etching of Si (10% KOH) to open the bottom view windows. The membranes were then released by wet etching of Si₃N₄ with hot phosphoric acid (H₃PO₄) at 180°C.



Fig. 3. Schematic representations of the wafer and the die layout of the differently shaped membranes.



Fig. 4. Schematic representation of the three general microfabrication steps used to process the specimens.

2.3. Experimental setup and test methodology

A schematic of the membrane deflection experimental setup is shown in Figs. 5 and 6. It consists of a nanoindenter, to apply load to the center of the membrane from

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Fig. 5. Side view of the MDE test showing vertical load being applied by the nanoindenter, P_V , the membrane in-plane load, P_M , and the position of the Mirau microscope objective.

the top, and a Mirau microscope interferometer positioned directly below the specimen to independently measure deflection through the microfabricated die window. A combined Nanoindenter and atomic force microscope (AFM) apparatus was used in this investigation to apply a line load to the center of the membranes. The typical experimental procedure can be described in three steps. The first step is to locate and characterize the membrane geometry by means of the optical and scanning capabilities of the AFM. Once the profile and surface geometry are stored, the wafer is moved to the test position to begin the second step. This is accomplished by means of an x-y translation stage with a positioning accuracy of 1 µm or better. The second step is the MDE itself. Parameters are set and a drift test is executed. Once the test criterion is reached, the membrane is loaded. Simultaneously, the aligned interferometric station is focused on the back surface of the film. The camera is then set to acquire digital images within a desired period of time. Force and displacement data are stored in the Nanoindenter controller PC, and full-field displacements are stored by acquiring monochromatic images. Prior to acquiring each set of images, the focus on the surface is updated to correct for the out-of-plane motion resulting from the downward displacement of the membrane.

The third step of the experiment is data reduction. Using the measured distance between fringes, obtained from the interferometer, and load and deflection data, obtained from the nanoindenter measurements, Cauchy stress and stretch are independently computed.

2.3.1. Alignment

In the MDE it is important to ensure that the membrane is loaded in a uniform manner to avoid spurious effects such as torsional forces and/or errors in true deflection. This is accomplished by ensuring that the nanoindenter line-load tip, membrane, and interferometer are all in alignment. The first step in the process is to align the membrane and the interferometer. The x- and y-axis rotational adjustments of the interferometer are tuned until all fringes disappear. Fig. 7(a) is an optical image of a membrane aligned with the interferometer. Some fringes are present due to the curved surface of





Fig. 6. Schematic of overall experimental setup. The top drawing is a topside view of the combined Nanoindenter/AFM rig and the bottom is a side view of the interferometer and sample stage.

the membrane. These are seen as the dark edges along the length of the membrane arm and the circles and semicircles on the contact area. It is important to eliminate fringes in the x-direction to remove errors as deflection occurs. The y-direction is also important since any misalignment will result in torsional stresses applied to the membrane.

The next step in alignment is to ensure the membrane is aligned with the nanoindenter line-load tip. In all likelihood the stage needs to be adjusted to achieve this



Fig. 7. Monochromatic images obtained from the interferometer showing: (a) a membrane perfectly aligned with the interferometer; (b) a membrane under load in which the nanoindenter tip, membrane, and interferometer are well aligned; note that fringes are developed evenly; and (c) a membrane under load in which the tip and membrane are out of alignment, fringes are not developed uniformly.

alignment. We have added two orthogonal cuts and countersink screws to be able to tilt the surface of the stage supporting the wafer. Deflecting the membrane by a modest amount in order to develop a few fringes checks alignment. If these fringes develop uniformly, noting that they will be slightly curved in the *y*-direction due to surface curvature, then the two are in alignment. If the fringes develop on an angle, then the stage must be adjusted to correctly position the membrane. The difference between an aligned and misaligned membrane is shown in Fig. 7(b) and (c). The proper direction in which the stage should be rotated around the x-axis is determined by observing the fringes on the contact area of the membrane. As the tip makes contact with one side of a misaligned membrane, point "p" in Fig. 8, the opposite side will develop fringes because the contact area, being significantly wider than the arms, will twist in the positive z-direction to bring it in contact with the tip and out of the plane of alignment with the interferometer. Thus, in the case of the schematic in Fig. 8, the stage must be rotated in the negative z-direction along the x-axis. The interferometer is then realigned with the membrane as described above. This process is repeated until fringes are developed uniformly.

2.3.2. Fringe development and measurement

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. The fringes are a result of phase differences of monochromatic light reflecting off the surface by traveling different path lengths to and from the membrane. This light is recombined with a reference beam of fixed path length.



Fig. 8. Schematic representation showing a nanoindenter tip and membrane out of alignment.

When the path length of the reflected light is a half of a wavelength, $\lambda/2$, out of phase with the reference beam they cancel each other resulting in a dark fringe. A fringe will occur at each $\lambda/2$ change in vertical height of the membrane. The relationship between the distance between fringes, δ , and vertical displacement is shown in Fig. 9(a). 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_1 . The average fringe distance, within the specimen gage region, is then obtained as $\delta = 0.5\lambda/\tan \theta_1$. From this information an overall strain, $\varepsilon(t)$, for the membrane can be computed from the following relation, viz.:

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

The above equation is only valid when deflections and angles are small. However, for larger angles a more comprehensive relation is required to account for the additional path length due to reflection off of the deflected membrane. This circumstance is examined in more detail in Fig. 9(b). The schematic representation shows the monochromatic pathways that generate two fringes next to each other. They originate from the objective plane, the exit plane of the Mirau objective lens, and then travel to and reflect off of the deflected membrane to finally return to the objective plane and recombine with the reference beam. The objective plane also represents the plane in which the digital image is captured. At large angles the difference in path length, η , due to the angled reflection becomes significant enough to affect deflection measurements. Using



Fig. 9. Schematic representation showing the relationship between distance between fringes (δ) and vertical displacement (a) and the correction in path length, η , to account for the angled reflection at large values of θ_1 (b).

geometrical relations, a correction factor can be found to accurately relate the measured distance between two fringes on the objective plane, δ' , with the associated distance between fringes at the membrane plane, δ , namely,

$$\delta = 4\delta' \cos^2 \theta_1$$
 and $\tan \theta_1 = \frac{\lambda/2}{\delta}$

The finite strain, s(t), can then be computed with this new δ , viz.

$$s(t) = \frac{\sqrt{\delta^2 + (\lambda/2)^2}}{\delta}$$

By acquiring images at periodic intervals, the deflection and strain can be mapped as a function of time. Fig. 10 shows a series of acquired digital images and fringe development on a membrane. Between 400 and 425 s, the membrane begins to exhibit a large localized plastic deformation, seen as discontinuous fringes, which eventually



Fig. 10. Composite of monochromatic images obtained from the interferometer at periodic intervals, from 0 s to fracture at 456 s.

led to failure. The frame at 456 s shows only the right part of the membrane; the left part is out of focus.

Although this measurement of strain is obtained in a local area on the membrane surface, it represents an overall strain for the entire gauged region and is thus, assumed to be uniformly distributed. This type of assumption allows the possibility of edge effects to enter the data, however, as mentioned in Section 2.1 the specimens were designed with specially tapered regions and sufficient length to remove these effects from the gauged region. In fact, tests performed on different specimen lengths provided identical Young's modulus, residual stress, and yield stress (see Section 4). In future studies, the authors will employ other strain measuring techniques, such as digital speckle correlation (DSC), to obtain localized strain data.

Another aspect of strain measurement stems from the fact that the nanoindenter also records the displacement history of the indenter tip. Much like the interferometer, this data also provides an overall strain for the specimen. Both methods yield strain values that match well for small angles of deflection. However, the nanoindenter method lacks some key aspects provided by the interferometer data. Primarily, the video image of fringe development clearly pinpoints the exact moment of contact between the nanoindenter tip and membrane. Determination of this point via the nanoindenter data is not so accurate with estimation errors resulting in distortions of residual stress and Young's modulus. The interferometer also allows verification that the membrane is loaded uniformly in its plane, see Fig. 7. Another advantage is that the onset of shear localization, see Fig. 10, and membrane failure are also observed.

3. Data reduction

The data recorded during the MDE test is in raw form and must be processed to obtain in-plane load and stress. The data obtained from a typical test is the Nanoindenter displacement in nanometers, load in milli-Newtons, and time in seconds as well as a video file of the fringe development recorded in Audio Video Interleave format (.avi).

3.1. Correcting the raw load signature

Reduction of the load data requires two steps. The first is the correction of the raw load obtained from the Nanoindenter. The second is the calculation of the membrane in-plane load. Besides the response of the membrane, the raw load signature is composed of other factors such as: stiffness of the gantry, stiffness of the support springs, changes in resistivity of the load coil, and thermal drift of the column. Of these factors the effect of the gantry stiffness is considered negligible since it is several orders of magnitude larger than the stiffness of the support springs or membrane. We also assume load coil resistance variability to be negligible. Thermal drift occurs when the gantry is expanding or contracting as a result of temperature fluctuations. The final effect on the measured load is due to the stiffness of the support springs. These springs support the nanoindenter column and have high stiffness in the direction perpendicular to the column, to avoid lateral deviations, while in the direction of the column their stiffness is approximately 100 N/m. Usually this effect is easily accounted for in standard nanoindentation, but in the case of membrane deflection experiments the load response of the membrane is one order of magnitude smaller than the springs' stiffness. As a result, a much more accurate procedure is needed.

The combined effect of the support spring stiffness and thermal drift factors results in a load response significantly larger than that of the typical membrane with the stiffness of the support springs being the largest contribution by far. In order to properly and accurately subtract these effects, they must be measured when the tip is not in contact with the membrane. This was accomplished by performing the test in air prior to deflecting the membrane. It was also determined that this air test needed to be performed at least four times to elicit an equilibrium and repetitive response from the



Fig. 11. Plot of raw load vs. displacement for air runs (thin lines) and membrane run (thick line). Runs 2–4 are close together and appear as one thick line.



Fig. 12. Load-displacement signatures for the corrected nanoindenter vertical load and the computed membrane in-plane load.

springs. A typical load-deflection signature of an air run is shown in Fig. 11. The response appears linear, but in actuality it is slightly nonlinear.

The air tests are immediately followed up by the actual membrane deflection. Loaddeflection signatures of the membrane and final air test are compared to determine the actual load response of the membrane. First, a polynomial representation of the load-displacement signature of air test is made. In the second step, the polynomial is directly subtracted from the membrane vertical load-displacement signature to remove thermal drift and spring stiffness effects. Typical membrane vertical load-displacement signatures of raw and corrected data are shown in Figs. 11 and 12, respectively. The load response of the membrane is dwarfed by the magnitude of the spring and drift effects. At the point of fracture the raw membrane load signature returns to follow that of the air test.

3.2. Conversion from nanoindenter vertical load to membrane load

The second processing to the measured vertical load response is geometric in nature, in that, the component of load in the membrane plane (P_V) , in the direction normal to the cross section must be computed from the load measured by the nanoindenter column (P_M) . A schematic of their relationship is shown in Fig. 5. As deflection increases, so does the angle θ . By using the deflection and the initial length of the membrane (L_m) , θ and P_M can be computed at any point during the test from the following equations:

$$\tan \theta = \frac{\Delta}{L_{\rm M}}$$
 and $P_{\rm M} = \frac{P_{\rm V}}{2\sin \theta}$.

Fig. 12 shows the difference in load signatures between the Nanoindenter (P_V) and membrane loads (P_M). The magnitude of the membrane load is significantly larger than that of the nanoindenter load. This disparity is a result of very small values of the sin(θ) at low deflections. At extremely low values of displacement this effect is also observed to magnify the scatter at the low end of resolution for the load cell, i.e., loads below 10 µN, as seen at the beginning of the membrane load signature. This scatter exists in the nanoindenter load as well, but is masked by the small magnitude of the measured loads. Upon fracture the loads of both signatures do not return to zero since half of the membrane is still engaged with the nanoindenter tip and is behaving as a cantilever. Once the actual load in the membrane is found, Cauchy stress, $\sigma(t)$, can be computed from

$$\sigma(t) = \frac{P_{\rm M}}{A}$$

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

4. Experimental data

4.1. Repeatability

An important feature of every new experimental method is repeatability in the measurements. To examine this issue, five experiments were performed on membranes of the same size within two wafers of different thickness (*T*), 0.5 and 1.0 µm. The load–deflection signatures from these thin film Au membranes are shown in Fig. 13(a) for $T = 0.5 \mu m$ and (b) for $T = 1.0 \mu m$. The membranes compared all have identical dimensions; length (L_M) = 372 µm, width (W) = 10 µm. The signatures of each thickness are significantly different and result from size effects that are clarified in part II of this article. At small magnitudes of displacement, in both plots, the membranes of each thickness exhibited identical behavior except for the region of very small displacements where the load cell is at its low end of sensitivity. This region corresponds to the early elastic deformation regime. As displacement increased, the membranes began to show evidence of plasticity and varying failure behavior. The strong agreement between the load–displacement signatures of the five membranes of each thickness indicates that



Fig. 13. Membrane load vs. displacement plots of five different identically sized membranes with film thickness of: (a) 0.5 μ m; and (b) 1.0 μ m.

the MDE procedure is a repeatable and reliable method to uniformly stretch, much like a direct tensile test, thin films and MEMS materials.

4.2. Stress–strain curves

Stress-strain curves for the membranes in Fig. 13 are shown in Fig. 14(a) for $T = 0.5 \ \mu\text{m}$ and (b) for $T = 1.0 \ \mu\text{m}$. Each membrane for both wafers followed identical elastic behavior with a measured Young's modulus of 53–55 GPa. As with the load-displacement signatures, the stress-strain curves differ greatly between each thickness. The measured modulus is significantly lower than the value of 78 GPa for bulk Au, however, values reported for thin film Au have varied from 30 to 78 GPa (Nix, 1989). The difference may result from the strong $\langle 1 1 1 \rangle$ texture exhibited by thin gold films (Harris and King, 1994,1998). The gold films used in this study also exhibit a strong $\langle 1 1 1 \rangle$ texture, see Section 3.1 in Part II of this article for further details. It is known that the Young's modulus of single crystal gold varies with orientation. For instance, $E_{\langle 111 \rangle} = 117$ GPa and $E_{\langle 100 \rangle} = 43$ GPa (Courtney, 1990). Thus, with $\langle 111 \rangle$



Fig. 14. Stress-strain curves of five different, but identically sized membranes with film thickness of: (a) $0.5 \ \mu m$ and (b) $1.0 \ \mu m$.

primarily normal to the film surface, the 53–55 GPa measured moduli are realistic. The uniform behavior of the membranes in the elastic regime lends further support to the repeatability and reliability of the MDE test.

Extrapolation of the elastic regime to the vertical axis provides an estimate of the membrane residual stress. This stress is for the suspended film after it has been released from its substrate. It should not be confused with the residual stress state of the film while on a substrate. In the case of the examined specimens the residual stress is tensile and in the range 10-12 MPa, see Fig. 14.

Yield stress was found to be approximately 170 MPa for all five of the 0.5 μ m thick specimens and 50–55 MPa for the 1.0 μ m thick membranes. The elastic regime of the 1.0 μ m thick specimens is expanded, to illustrate the variability in measured yield stress, in Fig. 15. It is known that the yield stress of polycrystalline metallic thin films strongly depends on their crystallographic texture (Thompson, 1993). In part II of this article we report on details of the films grain morphology and texture and their effect on yield stress. Here we just point to the fact that the scatter in yield stress, in the case of the 1.0 μ m thick membranes is 10% while the change in yield stress due to



Fig. 15. Expansion of the elastic region for Fig. 14(b).

a change in thickness from 1.0 to 0.5 μ m is about 340%! Likewise, the 1 μ m thick membranes show quite interesting post-yield behavior. Upon reaching their yield stress, each membrane began to undergo plastic deformation and quickly began exhibiting individual deformation behavior, especially for the 1.0 μ m thick specimens, followed by failure at varied stresses and strains. The 0.5 μ m thick membranes all exhibited sharp changes in stress and catastrophic failure. Some 1.0 μ m thick specimens showed catastrophic failure after a period of ductility while others showed a *progressive* reduction to zero stress. Also present in the plastic regime of the 1.0 μ m thick specimens are sharp undulations of stress indicating that plastic yielding happened in discrete manner. This feature is very much in contrast to the smooth hardening behavior of bulk metals.

4.3. Membrane size effects

Fig. 16 is a load-displacement plot for two different sized membranes. The solid circles (•) represent the same sized membrane presented earlier, length $(L_M) = 372 \mu m$, width $(W) = 10 \mu m$, thickness $(T) = 1.0 \mu m$, while the open circles (\bigcirc) represent a membrane of dimensions, length $(L_M) = 674 \mu m$, width $(W) = 20 \mu m$, thickness $(T) = 1.0 \mu m$. Both membranes have identical shapes and thickness, but their geometries are at different size scales. It should also be mentioned that they were processed on the same wafer. The load-displacement signatures of the two differ in some respects. The first is that the greater cross-section area of the larger membrane resulted in a larger load magnitude, as expected, as well as a greater displacement before failure occurred. Both membranes appear to have ruptured in a similar manner. The second noticeable difference between the two sizes is the load behavior at small displacements. In particular, the larger membrane exhibits a different loading behavior until a displacement of 15 μm where it abruptly changes its loading response to follow what would appear to be the extension of the smaller membrane load-displacement curve.

The stress-strain curves of the two different sized membranes are shown in Fig. 17. As in Fig. 16, the solid circles (\bullet) represent the smaller membrane and the open



Fig. 16. Comparison of membrane load-displacement signatures for two differently sized membranes; (\bullet) represents the membrane of dimensions length ($L_{\rm M}$) = 372 µm, width (W) = 10 µm, thickness (T) = 1.0 µm and (\bigcirc) represents the membrane of dimensions length ($L_{\rm M}$) = 674 µm, width (W) = 20 µm, thickness (T) = 1.0 µm.



Fig. 17. Comparison of stress-strain curves for the two membranes described in Fig. 16 using corresponding symbols.

circles (\bigcirc) represent the larger membrane. Both curves match rather well with identical Young's modulus of 53–55 GPa, identical yield stress of 55 MPa, and similar fracture stresses and strains. Some differences in the post-yielding behavior are clearly observed in the stress–strain plots. Further details of size effects in gold thin films are elucidated in part II of this work. Further results are presented in part II of this work.

5. Conclusions

A novel chip-level test has been presented to investigate mechanical properties of thin films used in the microelectronics industry as well as in the design of MEMS. The main advantages of the proposed test and methodology are their 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 can be used to investigate a variety of materials ranging from metals to ceramics including piezo-materials, shape memory alloys, ultra-nano-crystalline films, etc. By using the specimen ends as electrodes, direct measurement of current–voltage (I-V) diagrams resulting from the coupling between electrical and mechanical fields is feasible.

The MDE test results on Au films shows that micron-thick films can be tested by direct tension using a properly designed membrane specimen. The equipment employed was shown to possess the sensitivity and control required for such precise measurements. Our future work will involve testing Au films of various thickness and cross-section areas as well as other materials including membranes with oxide layers, simulating passivation. Investigations will also be performed to further study plasticity and fracture at the submicron scale.

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