Multi-layer metallic composites as strong electrode structures for MEMS

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1. ABSTRACT

If suspended membranes are to be used as support electrodes for piezoelectric films, they must be able to sustain large strains during operation. Metallic multilayered films can have strengths in excess of 1 GPa, making them possible support electrodes in piezoelectric micromechanical systems. In this study we compare the mechanical testing methodologies and film performance for two FCC-BCC multilayer film systems, Pt/Mo and Cu/Nb, as well as one tri-layer film of Cu/Nb/Ni.

2. INTRODUCTION

On the macro scale, tensile tests, compact tension tests, and four point bending are typically used to characterize plasticity and fracture behavior of materials. For evaluating materials properties in applications that require free standing MicroElectroMechanicalSystems (MEMS) there are several limitations to this type of testing. Difficulties in accurately measuring the loads and displacements on small scales and challenges in fabricating free standing test structures make it advantageous to use mechanical tests more suited to MEMS. While it is possible to carry out scaled-down versions of the macroscopic fracture tests on the micro and nanoscale, such as microtensile testing of FIB machined samples [1], and MEMS fabricated tensile, [2] fatigue, and fracture tests [3], some of the more common methods on this length scale are bulge testing [4] and beam element bending.

Bulge testing provides the opportunity to vary the stress state in the film betwen biaxial and uniaxial stress conditions by changing the aspect ratio of the pressurized membrane. Previous work has shown that it is possible to assess the elastic modulus and residual stress [5], the onset of plasticity, fracture, and fatigue [6] using this testing method. The strength in thin membranes for MEMS is of particular interest in developing acoustic transducers [7], an application in which significant monotonic strains applied to piezoelectric films significantly improves performance. One method to ensure that the films can support a large enough pressure is to use a silicon support layer, integral to many of the MEMS fabrication techniques. These support layers, while adding to the stiffness of the membranes, are often passive components. When using piezoelectric thin films at high strains (when strains due to stretching are significantly greater than those due to bending [8]) the support layers add no functionality. Therefore, there is interest in removing support layers and having a bottom electrode that operates as both the support layer as well as an active electrical component in these types of structures.

Platinum is often used as a bottom electrode for piezoelectric films, though it is possible to use Au with some modifications in the process parameters [9]. However, these metals have not been successfully fabricated into free-standing membranes due to the high residual stresses present in both the films after thermal processing and the subsequent piezoelectric films, which have residual tensile stresses on the order of 900 MPa. One current methodology to improve the strength of metallic films is to utilize nanoscale multilayers; the resulting films are stronger than either of the isolated component films [10]. In this paper, we will use two bilayer film systems Cu/Nb, and Pt/Mo and one tri-layer film system, Cu/Nb/Ni, to demonstrate the improvements in strength that can be achieved with multilayer film methodologies, document the onset of plastic deformation and report results of burst pressures for one of the film systems. The Cu/Nb film has been documented by other researchers and is used for baseline data, while the experimental Pt/Mo and trilayer films are being considered as alternative systems for higher temperature applications in oxidizing environments.

2. EXPERIMENTAL PROCEDURES

Both sets of films were grown using sequential DC magnetron sputtering. Pt/Mo films were deposited at a variety of thicknesses, while the Cu/Nb and Cu/Nb/Ni films were all kept at 20 nm per layer, with a total film thickness of approximately 2 μm. Films were deposited onto boron doped and oxidized silicon substrates, with a boron doped layer approximately 2 μm thick and a thermally grown oxide approximately 150 nm thick. The typical microstructure of the films consists of columnar grains separated by the interfaces, as shown in the scanning electron micrograph of the cross section of a film presented in Fig. 1.

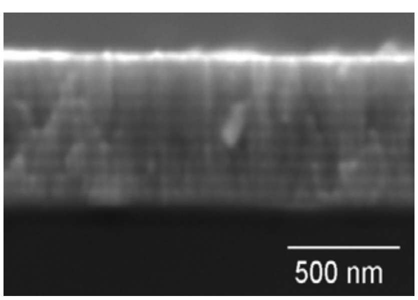

Fig. 1. SEM image of a cross section of a Pt/Mo multilayer film; 30 nm per layer. Distinct layers are visible (lighter layers are Pt) and columnar structures run through the film thickness.

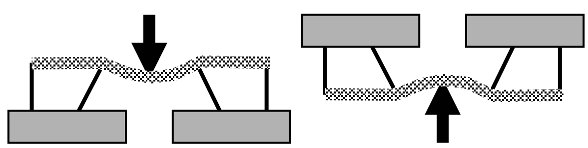

Fig. 2. Schematic of bulge testing free standing membranes to minimize film delamination. The Si die is glued to an Al puck (grey), and the chamber is either evacuated (left) or pressurized (right). Both geometries used for pressure-deflection testing, the right hand geometry was used for fracture testing

Bulge tested samples were fabricated on wafers patterned with windows 4 mm in side length (for the square geometry) and 10x2 mm (for the rectangular geometry). These structures were fabricated prior to metallic film deposition using wet chemical etching, leaving a boron doped Si and oxide support 2 μm thick [11]. After film deposition the samples were diced and the supporting oxide/Si film was etched using reactive ion etching in oxygen and CF_4 plasma to remove the entire support thickness. The etched freestanding film was then ready for bulge testing. Bulge testing was carried out in a custom built system. The die is mounted to an aluminum puck with water soluble glue such that the pressure is applied to the "top" of the films, using one of the two geometries shown in Fig. 2, this eliminates problems with delamination between the film and substrate. For assessing pressure – deflection relationships for the film the displacement at the center of the membrane was measured by using a Polytec OFV 511 scanning laser vibrometer, and the pressure was applied to the membrane using a Meriam Pressure/Vacuum variator, shown schematically in the left hand image of Fig. 2. For assessing the pressure needed to cause fracture the method shown in the right hand schematic of Fig. 2 was used, and pressure was applied using compressed gas; displacements were not measured in this case.

Nanoindentation was carried out using a Hysitron Triboscope using the nanoDMA™ attachment. This allows an assessment of the stiffness at various points in the loading sequence, such that modulus and hardness can be measured as functions of the indentation depth. All the reported hardness and modulus values made at depths between 5 and 10% of the total film thickness to avoid substrate effects.

3. RESULTS

Nanoindentation results of the hardness of the Pt/Mo films are shown in Table 1. The hardness of the Pt/Mo films, shown in Fig. 3 as a function of depth, demonstrates the need to assess mechanical properties at a depth which allows sufficient accuracy in the measurement, but still small enough to be uninfluenced by the underlying substrate. In general the hardness of the films increases as the thickness of the layers decreases; the softest films are those with Pt layers of 100 nm.

The pressure – deflection relationship, as shown in Fig. 4 for the Cu/Nb films, was used to measure the residual stress and elastic modulus of the film. The pressure – deflection relationship can be described using the geometry of the sample and materials properties [5]

$$P = \frac{C_1 \sigma_o t}{a^2} w + \frac{C_2 E t}{a^4} w^3 \qquad (1)$$

where P is the pressure, a the side length, t the film thickness, E is the elastic modulus, w is the center deflection, σ_0 is the residual stress, and C_1 and C_2 are constants which account for the geometry of the membranes.

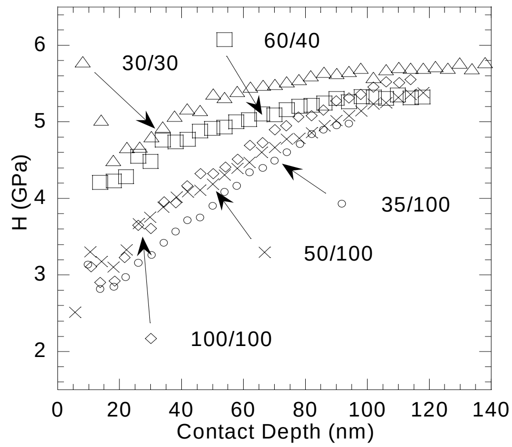

Fig. 3. Hardness as a function of depth for various thicknesses of Mo/Pt films; each curve is an average of 10 indentations. Error bars omitted for clarity.

Table 1. Hardness of multilayer films used in this study, measured at 5-10% of the film thickness.

Sample type	**Hardness (GPa)**
30nm/30nm Mo/Pt	5.36±-0.31
60nm/40nm Mo/Pt	4.91±- 0.27
35nm/100nm Mo/Pt	3.16±-0.57
50nm/100nm Mo/Pt	3.9±-0.34
100nm/100nm Mo/Pt	3.9±-0.34

The elastic modulus found from bulge testing is within 10% of that measured by nanoindentation at the appropriate 5-10% of the film thickness; the modulus measured by nanoindentation was 117±3 GPa while a modulus of 122 ± 13GPa was determined with bulge testing. The initial residual stress of the Cu/Nb films was approximately 135 MPa. The pressure – deflection relationship can be converted to the stress – strain ($\sigma - \varepsilon$) curve for the center of the membrane; for rectangular films this is done using

$$\sigma = \frac{a^2 P}{2wt} \qquad \text{and} \qquad \varepsilon = \frac{2w^2}{3a^2} \qquad (2,3)$$

where a is now the short side length of the rectangular membrane. However, the yield stress determined in this manner (see Fig. 4B), is not the highest stress in the membrane, as the stress concentration due to the corners of the membrane does lead to higher stresses. Therefore, a finite element simulation was carried out using ABAQUS for the different sample geometries tested in this study. The use of the FEM simulation to determine the stress concentrations in these membranes is similar to the methodology described by other researchers [12]. The resulting yield stresses do not correspond to a 0.2% offset, they are effectively sampling much smaller strains. These results are presented in Table 2.

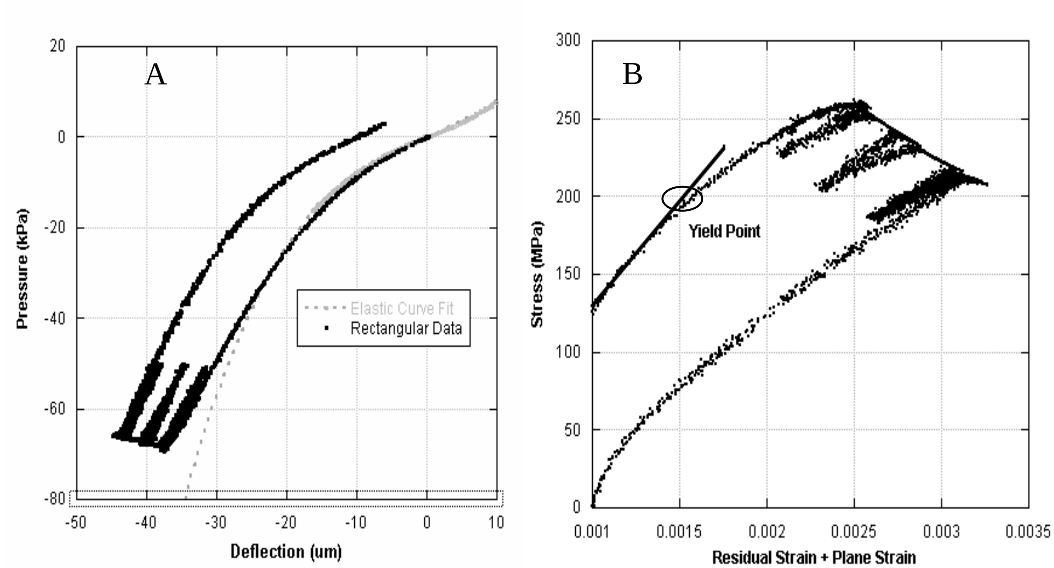

Fig. 4. A) Pressure-deflection curve showing plastic deformation. The specimen shown was loaded to a pressure of -67 kPa. The film was then unloaded slightly and reloaded several times, demonstrating elastic unloading after repeated plastic deformation. B) The representative stress vs. strain plot calculated from A.

Finally, Cu/Nb and Cu/Nb/Ni films 4 mm on a side with a total film thickness of approximately 2 μm were tested to failure using the pressurization scheme noted in Fig. 2. The burst pressures are presented in Table 2.

Table 2. Hardness, yield stress, and burst pressure for Cu/Nb and Cu/Nb/Ni films.

	Hardness (GPa)	Flow stress from H (MPa)	Yield Stress (MPa)	Burst Pressure kPa
Cu/Nb	3.4±-0.13	1200	421	269
Cu/Nb/Ni	2.3±.11	820	385	389

Burst tests of the Cu based films show that significant plasticity occurs during failure. Electron microscopy of the ruptured tri-layer film, Fig. 5, shows evidence of thinning along the crack path of the films. Small dimples on the fracture surface are additional evidence of plasticity that must be occurring during the failure of these structures.

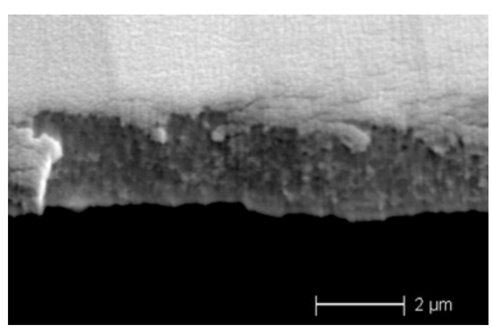

Fig. 5. Cross section of tri layer film showing dimples and thinning indicative of plasticity prior to fracture

A simulation of the dislocation behavior in the bulge test was carried out using a Multiscale Dislocation Dynamics Plasticity (MDDP) model developed at

Washington State University. [13,14] This model couples 3D discrete dislocation dynamics with continuum theory and finite element formulation, and accurately captures surface effects which have important implications in these thin film structures. A simulation of a bulge test (albeit it on a smaller scale than the experiment) is shown in Fig. 6 show an example of such numerical results for a bulge test of a pure Cu membrane. Figure 6a shows the deformed configuration of a square 0.3µmx3µmx3µm membrane which is clamped at all four edges and pressure is applied to its flat surface at a constant rate. The figure shows the contours of plastic strain that results from the evolution of dislocations, and results in the dislocation structure shown in Figure 6b. The predicted stress-strain curve for the particular case studied is shown in Figure 6c, showing a size-effect on the initial yield stress and strain hardening.

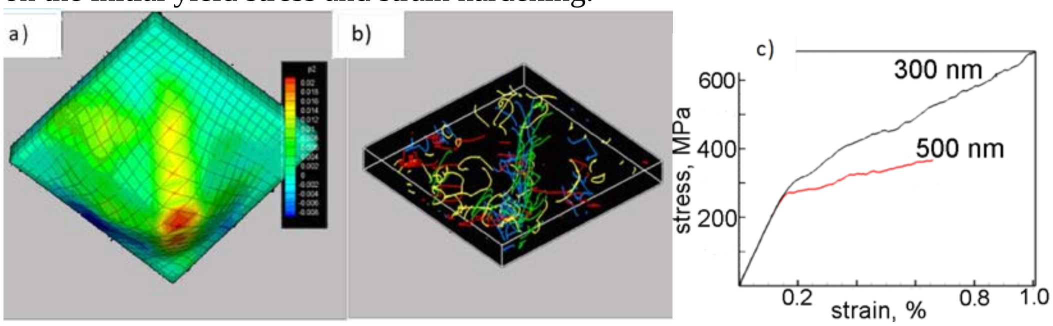

Fig. 6. MDDP results of a bulged Cu membrane with (a) deformed shape and strain contours (b) resulting dislocation structure and (c) stress strain curve for two different thicknesses.

4. DISCUSSION

The conventional method of converting hardness to flow stress for materials which show significant plastic deformation, is to divide the hardness by 2.8 [15]; this has been found to be similar to the flow stress at 8% strain. Even with this conversion the difference between the yield behavior and the flow stress suggests that small scale plasticity can be a major component in the deformation and fracture of these thin membranes.

The implication for the Pt/Mo films, when compared to the Cu film systems tested, is that the reliability and failure of freestanding metallic films operating at high strains requires an understanding of the plasticity mechanisms occurring in these structures. In particular, for oscillating structures (such as an acoustic transducer), the operating stress must be significantly less than what one would expect from following the classical assumptions regarding the flow stress and hardness. A conservative estimate of the stress required to initiate dislocation motion under biaxial tension would be on the order of 1/9th, and not 1/3rd, of the hardness of the films. The films likely show some strain hardening, based on the difference between the hardness and flow stress, though the non-uniform strain distribution may impact the actual magnitude of strain hardening. Additionally, as the softer Cu based films can sustain a substantially higher burst pressure, increasing the hardness alone will likely not result in an increased pressure to failure in the Pt/Mo system. As shown in the MDDP simulation and the bulge tests, work hardening in thin film metallic membranes is a potential issue that must be considered in regards to utilizing these structures in high strain MEMS devices, and will be the subject of further study.

5. CONCLUSIONS

The mechanical properties of metallic multilayer nanostructures (Pt/Mo, Cu/Nb, and Cu/Nb/Ni) have been assessed using both nanoindentation and bulge testing. The onset of plasticity in these structures is lower than one would expect from the conventional assumptions on hardness. Fracture occurs in metallic multilayers after some ductile deformation has occurred, suggesting that strengthening alone will not increase the failure strength of these materials in micromachined applications such as oscillating membrane structures. Strain hardening appears to be an issue in determining the ultimate pressure that can be supported by metallic membranes for MEMS; both simulations and experimental data suggest this to be the case.

6. ACKNOWLEDGEMENTS

The author wish to acknowledge the financial support of the Office of Basic Energy Science at the US Department of Energy under grant number DE-FG02-07ER46435. The assistance of Dr. A. Misra of Los Alamos National Laboratories for growth of the Cu/Nb and Cu/Nb/Ni films is greatly appreciated.

7. REFERENCES

[1] D. Kiener, W. Grosinger, G. Dehm, and R. Pippan, A further step towards an understanding of size-dependent crystal plasticity: In situ tension experiments of miniaturized single-crystal copper samples, Acta. Mater. 56, (2008) 580-592.

[2] R.L.Edwards, G Coles., W.N.Sharpe, Comparison of Tensile and Bulge Tests for Thin Film Silicon Nitride, Experimental Mechanics 44, (2004) 49-54.

[3] Muhlstein, C.L. ; Ritchie, R.O. High-cycle fatigue of micron-scale polycrystalline silicon films: Fracture mechanics analyses of the role of the silica/silicon interface, Intl. J. Fracture 120, (2003) 449-474.

[4]Y.Xiang, X.Chen, , J.J.Vlassak, Plane Strain Bulge Test for Thin Films, J. Mater. Res. 20, (2005) 2360-2370.

[5] J.J.Vlassak, W.D. Nix A New Bulge Test for the Determination of Young's Modulus and Poisson's Ratio of Thin Films, J. Mater. Res. 7, (1992) 3242-3249.

[6] O.N.Pierron, C.C.Abnet, C.L.Muhlstein, Methodology for low- and high-cycle fatigue characterization with kHz-frequency resonators, Sensors Actuators A 128, (2006) 140-150.

[7] D.J. Morris, D.F. Bahr, and M.J. Anderson, Displacement Amplification in Curved Piezoelectric Diaphragm Transducers, Sensors Actuators A 41, (2008) 262-265.
[8] U. Komaragiri, M. R. Begley, and J. G. Simmonds, The Mechanical Response of Freestanding Circular Elastic Films Under Point and Pressure Loads, J. Appl. Mech. 72, (2005) 203.
[9] M.C. Robinson, D.J. Morris, P.D. Hayenga, J.H. Cho, C.D. Richards, R.F. Richards, D.F. Bahr, Structural and electrical characterization of PZT on gold for micromachined piezoelectric membranes, Appl. Phys. A 85, (2006) 135-140.
[10] A. Misra, J.P. Hirth, R.G. Hoagland, Length-scale-dependent deformation mechanisms in incoherent metallic multilayered composites, Acta Mater. 53, (2005) 4817-4824.
[11] M.S.Kennedy, A.L.Olson , J.C.Raupp, N.R.Moody, D.F.Bahr, Coupling bulge testing and nanoindentation to characterize materials properties of bulk micromachined structures, Microsystem Techn. 11, (2005) 298-302.
[12] A. Reddy H.Kahn, A.Heuer, A MEMS-based evaluation of the mechanical properties of metallic thin films, J. Microelectromechanical Systems 16, (2007) 650-658.
[13]H.M.Zbib, and T. Diaz de la Rubia, A multiscale model of plasticity, Int. J. Plasticity 18, (2002) 1133-1163.
[14] H.M.Zbib, , T. Diaz de la Rubia, and V.A. Bulatov, A multiscale model of plasticity based on discrete dislocation dynamics, ASME J. Enger. Mater, Tech. 124, (2002) 78-87.
[15] D. Tabor The Hardness of Metals. Oxford Press (1951)