In-situ Mechanical Characterization of a 100 Nanometer Thick Freestanding Aluminum Film in TEM Using MEMS Force Sensors

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SUMMARY

We present a new experimental method for mechanical characterization of freestanding thin films with thicknesses on the order of nanometers to micrometers. The method utilizes MEMS force sensors and allows, for the first time, both quantitative and qualitative observation of materials response under uniaxial tension in SEM and TEM. Cofabrication of the specimen with force sensors eliminates problems related to gripping and force misalignment, and reduces the setup size. We demonstrate the method on a 100 nanometer thick freestanding Aluminum film inside a TEM. Preliminary results obtained indicate the applicability of the method as a tool for understanding the fundamentals of materials behavior in the micro/nano scale.

Keywords: Thin films, MEMS, Tensile testing

INTRODUCTION

Microelectronic and Micro-electromechanical systems (MEMS) experience external mechanical loads due to operational and environmental conditions, which causes the thin film components to fail as observed by cracking, delamination and formation of voids/hillocks[1]. Deformation and failure analysis in the micro/nano scales is far beyond mere interpolation from the bulk scale[2], which renders mechanical characterization of thin films an important step in the development of such systems.

At micro/nano size scales, materials behavior is influenced by interfaces and adjoining materials[3], and it is desired to characterize freestanding thin films. The uniaxial tensile test is popular for bulk materials, but it draws some challenges when applied to freestanding thin fims. These are (a) fabricating freestanding and residual stress free specimen, (b) gripping of the specimen, (c) aligning of the specimen in the direction of tensile force (even a slight misalignment may result in significant unaccounted flexural stress on the specimen, and invalidate the test by premature failure of the specimen), and (d) generating small forces (on the order of micronewtons) with high resolutions.

There are a few experimental techniques of uniaxial tensile testing of freestanding thin films in the literature. Hoffman[4] fabricated a 2 mm long, 150 μm wide and about 100 nanometer thick freestanding Aluminum film by evaporating the metal on a glass slide covered with a water soluble layer of Victawet, and then immersing it in water. The film was then glued to the grips of a nano-tensilometer with epoxy. Read[5] developed a method with force and displacement resolutions 200 micro-newtons and 20 nano-meters respectively. Ti-Cu-Ti multi-layer films with length of 700 microns, width of 200 microns, and total thickness of 1.2 μm were released from the substrate by wet etching. Huang and Spaepen[6] sputtered metal films with total thickness of 3 μm on 3 mm wide and 10 mm long glass slides, and then released the films by peeling them off from the substrate. A motor driven micrometer produced the strain with 0.002% resolution in the specimen, while a load cell read the stress with 0.1 MPa resolution. Rather than using wet processes (which can put large stress on the thin films) to fabricate the freestanding specimen, Haque & Saif[7] patterned 8 μm long, 2 μm wide, and 110 nanometers thick Aluminum film on a Silicon wafer by lithography, and used reactive ion etching technique to release the specimen. The specimen was tested with electrostatic comb drive actuators, with force resolution of upto 3 nanoNewtons.

In this study we present a microelectronic fabrication based technique to integrate a freestanding specimen with a MEMS force sensor. This integration ensures perfect alignment of the specimen to the loading direction. It also drastically reduces the experimental setup, thus enabling quantitative and qualitative in-situ experimentation in a TEM or SEM. Hitherto, such studies have been confined to qualitative nature, because the TEM straining stages do not control or measure stresses in the specimens.

DESIGN AND FABRICATION

The experimental setup consists of the integrated tensile test chip, and a mechanism for imposing displacement to the chip. In this study we used a motor driven straining stage on which the test chip is mounted. There are two holes on either side of the chip, which are designed to fit two mounting pins of a custom made TEM straining stage.

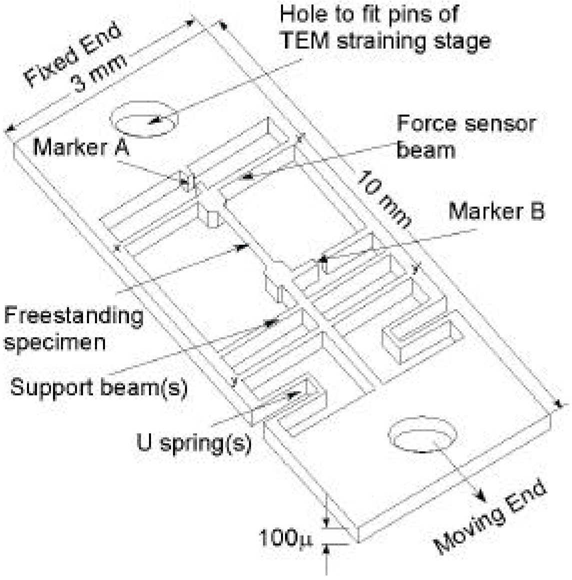

Fig. 1: Schematic of the tensile test chip

Figure 1 shows the schematic of the tensile test chip developed in the this study. The free standing thin film specimen is held at one end by a MEMS force sensor beam, which is allowed only in-plane deflections. The other end of the specimen is held by a set of supporting beams, which counter any off-axis force component on the specimen due to misaligned loading of the test chip. An FEM analysis shows that for the test chip used in this study, the rotation of the moving end of the specimen is six order less than the angle of misalignment between the specimen and the loading axis. Gripping of the specimen is due to the adhesion between the Silicon substrate and the specimen material. This eliminates the necessity of any extra gripping mechanism for the specimen. The U shaped springs maintain structural integrity between the fixed and the moving ends of the chip. The overall size of the chip is 10 mm x 3 mm x 100 microns. Figure 2 shows an SEM image of a fabricated test chip.

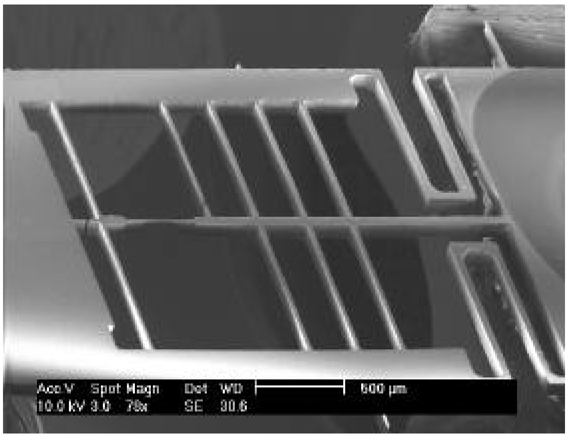

Fig.2: SEM image of a test chip.

Tensile force is applied on the specimen by imposing a displacement on one end of the chip, while the other end is held fixed. This displacement is transmitted to the force sensor beam by the specimen itself, causing a deflection in the beam. From the spring constant (k) and the measured displacement (δ) of the beam, the force (F) on the specimen is evaluated as $F = k\delta$. The spring constant is given by the equation below.

$$k = \left(\frac{24EI}{L^3} \right) \qquad [1]$$

where L is half the total length of the force sensor beam, E is the elastic modulus of the substrate material, I is the moment of inertia obtained from the beam cross-section. Since the force sensor is a beam made from single crystal Silicon along (110) crystal direction, the value of E is known accurately. More accurate value of the spring constant can be found by calibrating the force sensor beam with a Nanoindenter.

The force resolution depends on the spring constant of the force sensor beam, and the resolution of the measurement of δ. In this study, the spring constant value (k) is 102 N/m, and assuming 100 nanometers displacement can be read easily with the TEM, the force resolution is 10.2 µN. The two sets (A & B) of marker gaps in the chip read the displacements of both ends of the specimen. The readings of the marker A then gives the displacement δ of the force sensor beam. The relative displacements between markers A and B give the elongation of the specimen, which is subsequently used to compute the strain associated with the stress value. It is important to note that thin films may develop tensile residual stresses during film growth on a substrate[8]. The present experimental technique has the unique capability of measuring this stress in any pre-stressed specimen. Since one end of the specimen is attached to a deformable force sensor beam, any pre-stress in the specimen can be detected by the displacement in the force sensor beam (by reading the marker gap at A in figure 1) just before the experiment, and after the specimen fractures.

Test Chip Fabrication:

The process steps for fabricating the tensile test chip are as shown in figure 3.

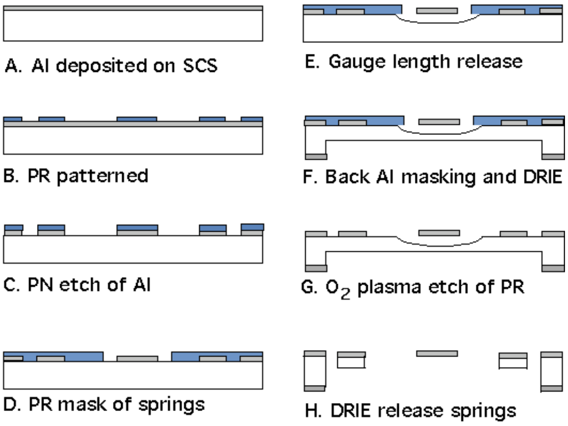

Fig. 3: Fabrication process schematic.

A. Aluminum film is sputtered on a Silicon wafer with a thickness of 100 nanometers.
B. The test chip, as shown in figure 1, is patterned on the wafer by photolithography.
C. The pattern is transferred to Aluminum by wet etching of the film.
D. The entire test chip area, except the specimen gauge length, is masked with photoresist by lithography.
E. The specimen is released from the substrate by isotropic, reactive ion etching using SF_6 gas.
F. Aluminum is deposited on the backside of the wafer, and an etch mask covering the entire test chip area is patterned by lithography, and subsequent etching of Aluminum. The wafer is anisotropically etched from the backside with Deep Reactive Ion Etching (DRIE).
G. The remaining photoresist on the frontside of the wafer is ashed off by O_2 plasma etching.
H. The wafer is etched anisotropically from the frontside with DRIE. The structural beams and the force sensors are fabricated in this step

Experimental Setup:

The tensile test chip is designed to fit in a TEM straining stage where one end of the chip is pulled by a motor. The stage is custom made for a JEOL 4000 TEM equipped with an environmental cell. The test chip is mounted on the stage by meshing the holes on the two ends of the chip with the two pins of the stage. This is shown in figure 4. The pins are threaded, so that the chip is secured by washers and nuts.

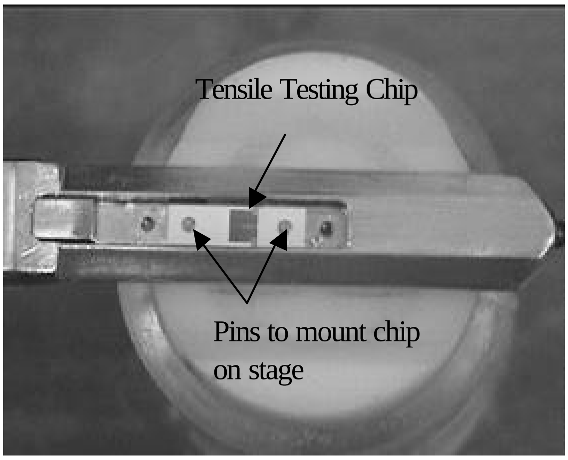

Fig. 4: The test chip mounted on a TEM straining stage.

EXPERIMENTAL RESULTS

We demonstrate the applicability of the present technique by fabricating and testing a 100 nanometer thick, 400 microns long, and 20 microns wide freestanding Aluminum specimen. Figure 5 shows the low magnification TEM image of the test chip. The experiment was conducted by switching between low and high magnification modes to read the displacements in the marker gaps and also to observe the microstructural aspects of the deformation process.

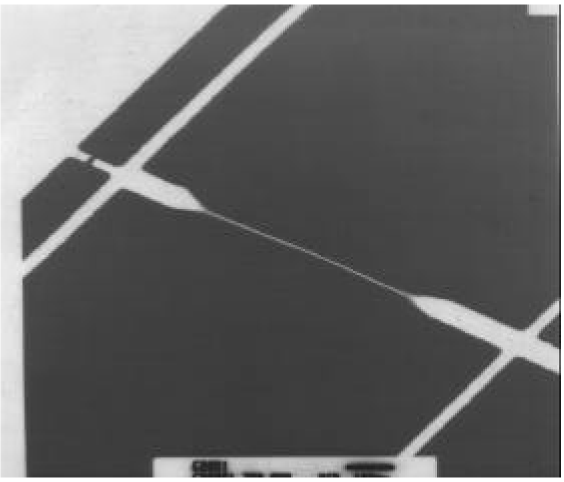

Fig. 5: The tensile test chip showing the specimen and the marker gap A inside TEM (60x).

The TEM images of the film show that the average grain size in the film was 60 nanometers. No crystal imperfections such as dislocations and voids were observed in the specimen. Upon loading, the specimen fractured before any useful data could be obtained. This premature failure can be attributed to preexisting cracks in the specimen due to high residual stress. Figure 6 shows the SEM image of the fractured specimen.

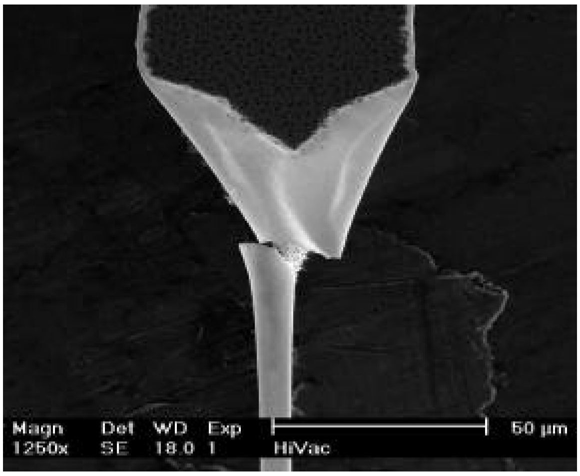

Fig. 6: SEM image of the fractured specimen.

CONCLUSIONS

We present a new technique for uniaxial tensile testing of thin films with the following features:

1. It allows testing of any freestanding single or multilayered thin films that can be grown/deposited on Silicon.
2. It addresses the problems of pre-loading, gripping, and misalignment of the specimen, making the test truly uniaxial.
3. It allows both quantitative and qualitative in-situ testing inside a TEM or SEM chamber.

We applied the technique to test a freestanding 100 nanometer thick Aluminum film inside the TEM. While we were not able to obtain enough data to extract mechanical properties, we can conclude that at this size scale, Aluminums films are virtually free from crystal imperfections.

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