Investigation of the Deformation Mechanics in Nanoindenter Deflected Freestanding Submicron Gold Thin Films

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ABSTRACT

The Membrane Deflection Experiment (MDE), developed by Espinosa and co-workers[1, 2], was employed to perform the microscale tensile testing on freestanding gold thin films. Gold films with varying thickness of 0.25 to 1.0 µm were deposited by both E-Beam evaporation and sputtering techniques. High-resolution SEM, including electron-backscattered diffraction (EBSD), was employed to provide a crystallographic analysis including grain orientation maps of the studied films. The Young's modulus of gold deposited by E-Beam evaporation was measured consistently in the range of 55-60 GPa while 68-72 GPa for the sputtered films. Plastic yielding of the e-beam and sputtered films was contrasted due to varying microstructure of each deposition technique, which appears to assert a measure of control on the deformation mechanics. An analysis will be presented on the effects of microstructure and crystalline texture.

Keywords: thin film, elastic modulus, micro-tensile testing, EBSD

1 INTRODUCTION

Thin film materials have been widely employed in microelectronic device and Microelectromechanical systems (MEMS) for many years. Accurate knowledge of the mechanical properties of thin films employed in these devices is critical to predict their performance and reliability. Therefore, many characterization methods have been developed to determine its values, including nano-indentation test, micro-beam bending test and bulge test etc[3-6]. In this paper, The Membrane Deflection Experiment (MDE), developed by Espinosa and co-workers [2, 7], was employed to perform the microscale tensile testing. The mechanical response of thin films depends on many factors. As the film dimensions begin to approach the scale of the material microstructure features, the material mechanical properties begin to exhibit a strong dependence on the microstructure and specimen size as well.

The microstructure of thin films strongly depends on the deposition process used. Characteristics such as grain size and shape distribution, and crystalline orientation are affected by the conditions under which grain nucleation, growth, coarsening, and thickening occur [8]. Two processing techniques are used to modulate the microstructure of the thin film specimens tested.

2 EXPERIMENTAL DETAILS

2.1 Sample Preparation

Gold films were deposited onto (100) Si wafers by both Ebeam evaporation and Sputter deposition. CHA Mark 50 evaporation system and Denton Vacuum Discovery 18 sputter system were employed respectively. The gold target was purchased from Electronic Space Products Inc. with purity of 99.99% for sputtering and kamis with purity of 99.99% for Ebeam. The back ground vacuum was achieved at $1x10^{-6}$ Torr for sputtering and $1x10^{-7}$ Torr for Ebeam. DC sputter power density of $4.5w/cm^2$ and Ar flowing rate of 25sccm with the process pressure of 5mTorr were employed for all sputtered films. Thin film thickness was controlled by sputtering time and characterized by TENCOR alpha-step 200 profilometer. Deposition rates are 4.0A/s for Ebeam and around 10A/s for sputtering. A thin layer of Ti was always deposited as an adhesion promoter prior Au film deposition without breaking the vacuum.

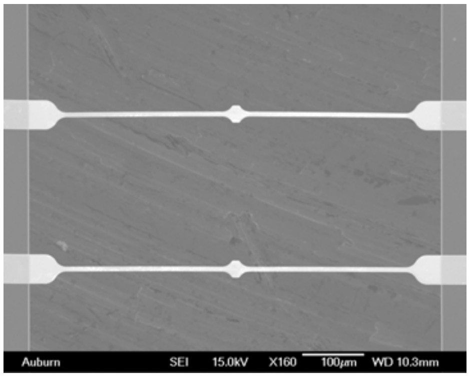

Figure 1: SEM image of one micro fabricated MDE Sample.

MDE test specimens were fabricated on double side polished 4 inch (100) single crystal silicon wafers. Barrier layers of Si_3N_4 were deposited on both sides by LPCVD. Bottom windows were opened by anisotropic KOH etching. The freestanding membranes were finally released by plasma etching of Si_3N_4 and Chemical etching of Ti. The details of the fabrication procedure are described in [2].

Figure 1 shows a SEM image of the Au membranes with half length of 250um, width of 5 um and thickness of 500nm.

2.2 Microstructure Characterization

The grain structures of the gold thin film deposited on Si/Si_3N_4 substrate with various thicknesses were characterized using the field emission scanning electron microscope (FESEM JSM -7000F) from JEOL. Uniform grain sizes of the gold films are clearly developed in each case as seen in figure 2. An increase of grain size is observed with the increase of film thickness, which may result from higher thermal energy induced and larger number of Au atoms available for aggregation for thicker film. Because the thicker the film was deposited, the longer processing time was needed and the higher temperature on substrates was developed.

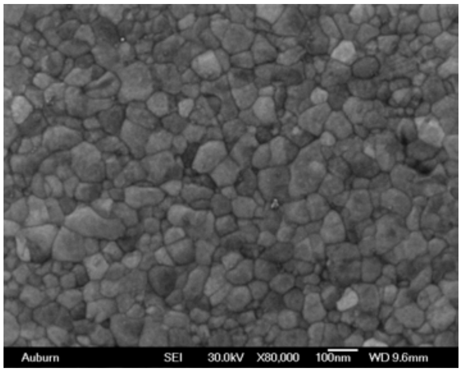

Figure2: FESEM image showing the grain structure of sputtered film with thickness of 250nm.

Crystal structures of gold films were characterized by Rigaku X-Ray Vertical Diffractometer with Cu $K\alpha_1$ radiation; and HKL electron backscattering diffraction (EBSD). A very strong (111) preferred orientation polycrystalline structure of both the sputtered and Ebeam Au film was observed. Figure 3 is the micro diffraction result from EBSD, which confirmed the preferred <111> texture of the Au films.

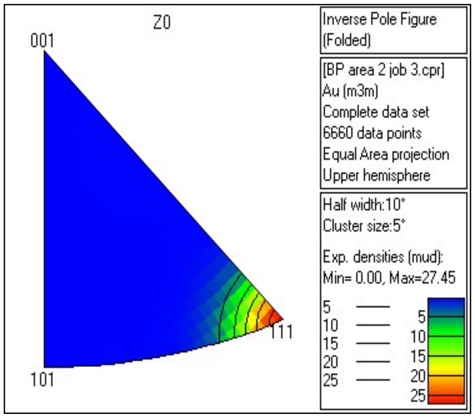

Figure 3: Micro-diffraction results showing a preferred <111> texture normal to the film surface.

2.3 Micro-tensile Testing

The membrane deflection experiment set-up consists of a MTS Nanoindenter system equipped with a wedge tip, to apply line load to the center of the membrane from the top; A Mirau microscope interferometer integrated with a five axis manipulation component, a CCD camera onto our customized working stage, positioned directly below the specimen to independently measure deflection through the microfabricated die window, as shown in figure 4. Calibration and alignment between nanoindenter tip, freestanding membrane and Mirau objective were performed prior to the testing to ensure that the membrane was loaded at the center area and perpendicular to the plane of membrane.

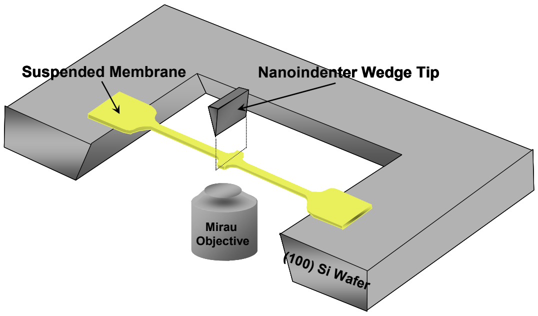

Figure4: Schematic of the membrane deflection experiment set up.

The load data directly obtained from the Nanoindenter must be reduced to the load in the plane of membrane, which is found as a component of the vertical Nanoindenter load by the following equations:

$$\tan\theta = \frac{\Delta}{L_m} \qquad (1)$$

$$P_M = \frac{P_V}{2Sin\theta} \qquad (2)$$

Where θ is the angle of rotation, 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, parameters shown in figure 5. Once P_M is obtained, the stress, σ(t), can be computed from:

$$\sigma(t) = \frac{P_M}{A} \qquad (3)$$

Where A is the cross-sectional area of the membrane in the gauge region. And the strain is calculated as:

$$\varepsilon = \frac{\frac{L_M}{Cos\theta} - L_M}{L_M} = \frac{1}{Cos\theta} - 1 \tag{4}$$

Stress-Strain curves then can be generated based on these information provided.

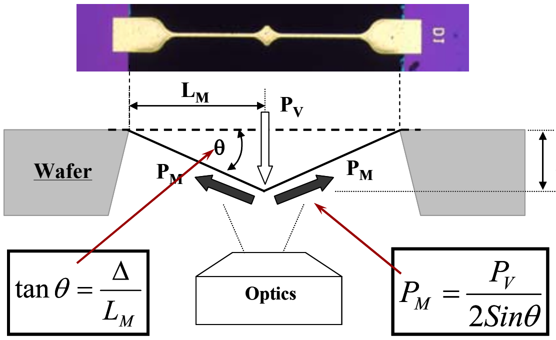

Figure 5: Schematic of the side view of membrane deflection experiment set up.

3 RESULT AND DISCUSSION

The stress strain curve was generated from the interpretation of the nanoindenter data. Mechanical properties including Young's modulus and yield stress can be extracted from the curves. Figure 6 is the 5 stress strain curves obtained from one group of 5 Au film membranes with thickness of 500nm, width of 10µm and length of 300µm. Young's modulus of 69-72 GPa were extracted with high repeatability from the curves.

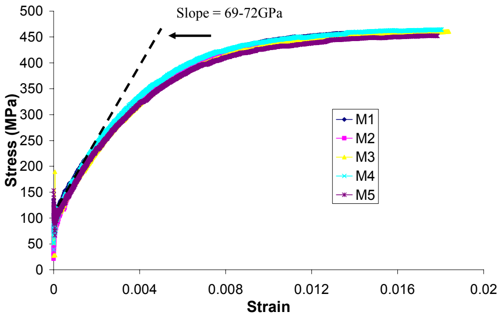

Figure 6: The stress strain curve for one group of 5 Au membranes (Thickness=500nm, Width=10µm, length=300µm).

The modulus measured is in the range of 68-72GPa for the sputtered films with thickness changing from 250nm to 1000nm and 55-60Gpa for Ebeam films. Figure 7 summarized the Young's modulus values of Au films with different thickness. The differences in Young's modulus values between the sputtered films and Ebeam films are believed to relate to their deposition mechanisms, which determine the grain structure and film crystalline texture developed during film growth [4]. The grain structures characterized by SEM shows a difference in grain size change with film thickness for sputtered and Ebeam film as table 1 summarized, also, a more uniform grain distribution was observed in sputtered film than in Ebeam film. Although both the sputtered film and Ebeam film have shown a strong preferred (111) orientation texture, the fraction of the preferred orientation was found higher in Ebeam film than sputtered film.

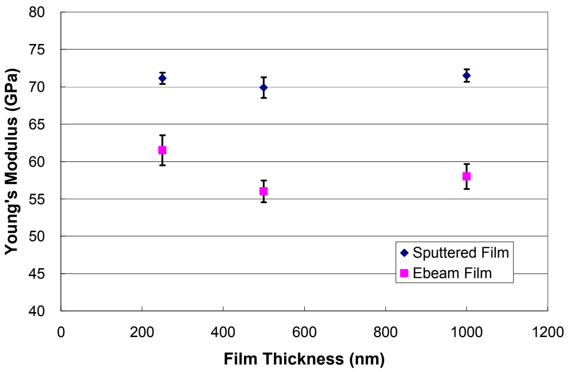

Figure 7: Measured Young's modulus of sputtered and Ebeam Au films with various thickness.

The Young's modulus measurement for both sputtered and Ebeam films only exhibits a dependence on deposition technique, However, there is a strong dependence of film thickness on the yield strength as well as the residual stress for both sputtered and Ebeam films. The yield stress was increased with thickness decreasing as shown in Figure 8, yield stress as high as nearly 650MPa for the thinnest films while around 350MPa for the 1000nm films, which is in accordance with the Hall-Patch theory as summarized in Table 1, the grain size increases with the film thickness increase.

Thickness	Sputtered Film	EBeam Film
250nm	60nm	25nm
500nm	100nm	50nm
1000nm	150nm	80nm

Table 1: Grain Size Measurement on Sputtered and Ebeam Au thin films.

Residual stress measured from the tests also shown the thickness effect as illustrated in figure 9. An average residual stress of 150 MPa for 250nm thick film while under 20MPa for 1000nm films. Figure 8 and figure 9 presented as following are the result for sputtered films, a similar film thickness dependence of yield stress and residual stress was observed in Ebeam film as well.

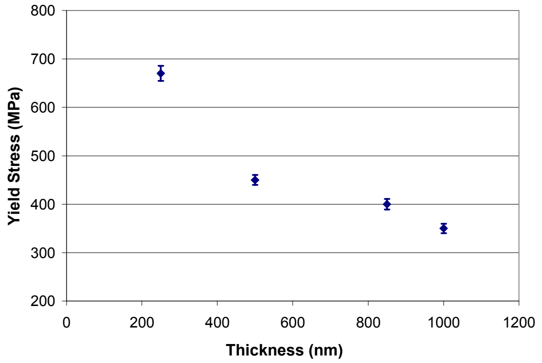

Figure 8: Measured Yield stress values of sputtered films with various thickness.

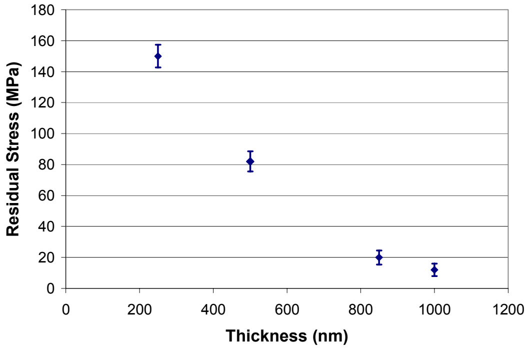

Figure 9: Measured Residual stress values of sputtered films with various thickness.

4 CONCLUTION

Thin gold films with varying thickness of 0.25 to 1.0 µm deposited by both E-Beam evaporation and sputtering techniques were carefully and comparatively studied. A difference of nearly 10Gpa in Young's modulus values measured for sputtered and Ebeam films indicate the strong relationship between the deformation mechanics and the microstructure determined by the various deposition techniques of the films. Effect of film thickness on Young's modulus, yield stress and residual stress has been analyzed. Further investigation on the grain structure and film crystalline structure effects on both elastic and plastic deformation mechanism of submicron scale are needed.

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