Effect of Strain-rate on the Mechanical Behavior of Pt-Films for MEMS

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1 ABSTRACT
A method to measure the mechanical behavior of Pt thin films was developed. It employs high magnification optical microscopy and Digital Image Correlation to extract sub-micron (100 nm local displacement resolution) deformation fields in free-standing thin films and calculate their elastic/plastic properties as a function of strain rate. In this study, the specimen gauge length and thickness were 1000 μm and 400 nm, respectively, and the gauge width varied between 100 μm and 200 μm. The micron scale specimens were loaded using a custom-made piezoelectrically driven micro-tension system and the applied nominal strain rates were between 10⁻⁶ to 10⁻³ s⁻¹. The stress-strain diagrams showed a linear elastic regime up to the yield stress, with elastic modulus E = 178.3 ± 6.2 GPa.

2 INTRODUCTION
Mechanical characterization of thin metallic films is of particular importance for microelectronic, data storage, and micro-electro-mechanical systems (MEMS) materials that are subject to thermomechanical loads. A number of factors, i.e. device size/geometry, grain size and texture, local diffusivities and grain boundaries, as well as the fabrication process, applied strain rate and temperature may influence the effective mechanical response of nanostructured thin films. Pt is among the materials commonly used in MEMS applications, and it is employed in RF-MEMS switches, antennas, and filters and, in general, it is characterized by good electrical conductivity, thermal stability, ductility, and corrosion resistance [1]. When integrated in active MEMS devices, Pt thin films experience mechanical and thermal loads due to operational and environmental conditions, which may affect their mechanical integrity. To ensure long-term reliability and performance, the mechanical properties of Pt thin films must be investigated as a function of device size and be related to its underlying microstructural (grain) details.

Typical experimental techniques for assessing the mechanical behavior of free-standing thin films are uniaxial tension tests [2,3,4,5,6], bulge tests [7], surface Brillouin scattering (SBS) [8], etc. Due to intrinsic assumptions in each experimental method, experiments have shown that the results are not always consistent among different laboratories. Among the aforementioned methods, uniaxial tension is the preferred method because it facilitates the straightforward calculation of material properties from the experimental data. In uniaxial tension tests, uniform tension is imposed on a thin film (plane stress condition), so that uniform strain is developed in the specimen gage section and the failure strength of microscale specimens is directly determined.

In 1972, Platinum Metals Review[9] reported the elastic modulus of bulk Pt as E=172 GPa, its tensile strength as σf =124 MPa and a hardness value (HV) of 40-42. Using a resonance technique, Jurgen et al. [10] reported that the elastic modulus of Pt steadily decreases with increasing temperature. Specifically, it decreased from E= 164.6 GPa at room temperature to E= 132.7 GPa at 900 °C, while its Poisson’s ratio was approximately constant, ν = 0.40, over the entire temperature range. Salvadori et al. [11] used cantilever resonance experiments and beam theory analysis to obtain the Young’s modulus of Pt thin films as 139 ± 2.7 GPa. In their study, Pt was uniformly coated on Atomic Force Microscope (AFM) cantilevers with thicknesses between 18 nm and 73 nm. Finally, using wafer
curvature and nanoindentation Seungmin et al. [12] measured E = 163 GPa from 200 nm thick films. Their results were compared to flow stress calculations based on existing dislocation motion models and good agreement was established.

However, to date no systematic uniaxial tension tests of Pt thin films have been reported. Due to the small specimen size, handling, aligning and gripping of microscale specimens, as well as measuring small loads and displacements tension tests at the micron scale are not a trivial task. In this paper, a full-field method to obtain the mechanical property of Pt thin films is presented. Strain measurement was conducted by optical microscopy and Digital Image Correlation (DIC) analysis [13]. Local deformation fields were extracted, and the elastic modulus and elastic/plastic properties were derived.

3 EXPERIMENTAL PROCEDURE
3.1 SPECIMEN PREPARATION
Thin film specimens were fabricated at the US Army Research Laboratory's Specialty Electronic Materials and Sensors Cleanroom Facility. The specimens were fabricated on (100) silicon substrates. Metal thin films were deposited via DC magnetron sputtering using a Unaxis Clusterline 200 multi-chamber cluster tool. A base layer of titanium (~160 Å) was first deposited to serve as an adhesive layer for the platinum (5000 Å) thin film. The deposition conditions are outlined in Table 1.

<table>
<thead>
<tr>
<th>Material</th>
<th>Ar (sccm)</th>
<th>O2 (sccm)</th>
<th>Pressure (mT)</th>
<th>Temp (ºC)</th>
<th>Power (W)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ti</td>
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<td>0</td>
<td>5</td>
<td>50</td>
<td>1000</td>
</tr>
<tr>
<td>Pt</td>
<td>50</td>
<td>0</td>
<td>5</td>
<td>50</td>
<td>500</td>
</tr>
</tbody>
</table>

Table 1: Deposition conditions

The process to define the critical components of the tensile specimens began by patterning the Ti/Pt thin films with argon ion-milling. This process started by patterning ~2.0 μm layer of Clariant AZ 5200 series resist cured at 110ºC. This same photoresist was used throughout the fabrication process. The photoresist was patterned using a Karl Suss MA/BA-6 mask aligner and subsequently developed using a Karl Suss ACS 200 resist cluster tool. Following inspection of the photoresist pattern, the wafer underwent a descum procedure in an oxygen plasma to remove any remnant resist. The descum process was completed following each photoresist patterning procedure. Next, the resist underwent an ultra-violent (UV) curing process at 200ºC for 30 seconds to reduce the potential of platinum being re-deposited during the ion-milling process. The UV cured resist has a hard crust layer that forms thereby minimizing the potential of etched non-volatile species from being embedded in the resist. The exposed metal layers were removed using a Commonwealth Scientific argon ion milling. The angle of incidence during the milling was manually controlled to 85° for the bulk of the procedure. To minimize re-deposition on the sidewalls of the resist, the final 30 seconds were milled at an angle of 50°. After the ion-mill, the resist was ashed in an oxygen plasma.

In the next process step, markers and device labels were deposited with electron beam evaporation with a CHA evaporator. The labels consisted of 200 Å titanium / 2000 Å gold. After deposition, the features were patterned with liftoff by placing the wafers into a heated bath at 85ºC of Baker PRS-3000 positive resist stripper. The wafers were soaked until all regions with unwanted metallization were detached from the wafer surface. Afterwards, the wafers were rinsed with de-ionized water. To ensure the metal features have clean edges (i.e. to remove wings), the wafers undergo 10 seconds of ultrasonic cleaning in de-ionized water.

The samples were prepared for die separation by coating the front surface of the wafer with photoresist. Afterwards, the wafer undergoes a dicing process using a DISCO dicing saw. In this procedure, the saw cuts thru half the wafer thickness (500 μm). This insures that the wafer stays intact for the remaining process steps.

Following the dicing process, the resist is removed using a acetone followed by methanol, isopropyl alcohol, and de-ionized water. To create free-standing tensile specimens, the silicon substrate is removed isotropically with xenon difluoride from underneath each specimen. Prior to the release etch, the wafer was exposed to a 20 second
reactive ion etch using CF$_4$, CHF$_3$, and He to remove a surface oxide that developed on the exposed silicon surfaces. In the release etch, the silicon was removed using a non-plasma, dry process with a combination of XeF$_2$ (2 T) and N$_2$ (20 T) gases with an etch cycle of 20 seconds using a Xactix Xetch. Each etch was completed with approximately several hundred etch cycles because of the loading on the system from all of the exposed silicon on the wafer. Following the release, individual die containing 6 tensile specimens were separated from the wafer and prepared for testing.

3.2 EXPERIMENTAL APPARATUS

Figure 1 shows the experiment layout built for this study. A tension-compression load cell with capacity 50 grams and $10^{-2}$ gram accuracy was used to measure the load carried by the Pt thin films. The specimens were loaded by an open-loop piezoelectric (PZT) actuator with a total travel of 60 microns and digitization resolution of 6 nm. The free-standing Pt thin films attached to Si substrates (Figure 2) were mounted on the metal specimen holder directly attached to the load cell. A flat glass grip mounted on tilt correction fixture on the PZT actuator was used to grip the specimen paddle (Figure 2). The flat glass grip ensured plane stress loading of the tensile specimen and its direct orientation. The UV-curable adhesive was deposited on its surface and used to fix the large specimen grip [5,14].

The tension tests were conducted to measure full-field strains from the surface of the specimens. The test apparatus was installed under an optical microscope and images were recorded at 200× with the aid of a long working distance objective. A CCD camera with 1024 × 768 pixel$^2$ resolution and 15 fps maximum frame rate was used to image the specimen gauge section at domain sizes of 480 × 360 μm$^2$. In order to compute specimen strain with the aid of DIC, a random speckle pattern was generated on the specimen surface by dispersion of silicon powders with average particle size of 1 μm (Figure 3).
The specimens were first loaded to 3000 nm of cross-head displacement and then unloaded by 2700 nm at constant displacement rate to obtain the loading/unloading stress-strain curves within the material elastic regime. Subsequently, reloading was assumed at the same rate until specimen fracture. Different displacement rates were applied to obtain nominal strain rates from $10^{-6}$ s$^{-1}$ to $10^{-3}$ s$^{-1}$. This strain rate range can be extended to faster and slower rates and it is only limited by the imaging rate of the CCD camera. The engineering stress was measured from the load cell data while the engineering strain was determined by DIC with 100 nm accuracy. The experimental data were used to obtain the thin film material effective elastic/plastic properties following the approaches described in [15].

4 RESULTS AND DISCUSSION

Figure 4 shows a typical stress/strain curve of a Pt film including the initial loading-unloading segments at a strain rate of $7.1 \times 10^{-3}$ /s. The elastic moduli computed in the initial loading, unloading and final reloading segments of the curve were 174.61 GPa, 173.09 GPa, and 173.34 GPa, respectively. These values are consistent with the reported bulk Pt values. The yield and ultimate tensile strength of this thin film were 1200 MPa and 1800 MPa, respectively. Figure 5 shows the contour of axial displacement (U) in $\mu$m as computed by DIC analysis.

Collective $\sigma$-$\varepsilon$ curves are plotted in Figure 6 at strain rates $10^{-6}$ - $10^{-3}$ /s. The elastic modulus was not affected by the strain rate and averaged 178.3 $\pm$ 6.2 GPa. It should be noted that the value of the Young's modulus is proportionally affected by the value of the film thickness that was accurate within 5%. The nominal film thickness measured during fabrication was 500 nm and the measured thickness after specimen release, as determined with a Scanning Electron Microscope (SEM), was 400-420 nm. The tensile strength was not significantly affected by the strain rate, which is consistent with the behavior of other FCC metals at room temperature. It changed with strain rate within 2% of its average value and, as expected, it increased with increasing loading rate. The ultimate tensile strength at strain rates $3.6 \times 10^{-3}$ /s, $7.0 \times 10^{-4}$ /s, $8.3 \times 10^{-5}$ /s, and $8.3 \times 10^{-6}$ /s was 1830 MPa, 1800 MPa, 1820 MPa, and 1790 MPa, respectively. These values are significantly higher than the bulk values and they are potentially the consequence of limited defect density in the tested films.

The ductility of Pt films was subject to strain rate sensitivity: as the strain rate increased, the ultimate strain increased accordingly. The highest ultimate strain was 4.3% at $8.3 \times 10^{-5}$ /s and 4.2% at $8.3 \times 10^{-6}$ /s, while at the low strain rate of $3.6 \times 10^{-3}$ /s the ultimate strain was 3.2%. It is expected that further decrease in the loading rate will allow for enhanced ductility as a consequence of substantial material creep at stresses larger than the yield strength. At $3.6 \times 10^{-3}$ /s strain rate, the proportional limit of thin film was 1040 MPa, while at lower strain rate tests
at $8.3 \times 10^{-5} /s$ and $8.3 \times 10^{-6} /s$, the corresponding proportional limits were equal to 1020 MPa and 980 MPa, respectively. The relatively small change in the material proportional limit due to loading rate difference indicates limited creep and temperature dependence of material yield. Furthermore, large proportional limit values contribute to long-term material stability at high stresses and significant thermal loads. Additional experiments are under way to derive the material response in a broad range of loading rates. Finally, the properties reported here did not show dependence on the specimen width that varied between 100-200 microns, while the specimen thickness and grain size were constant.

![Stress-strain curves of Pt thin films at subject to tensile loading at different strain rates](image)

**Figure 6:** Stress-strain curves of Pt thin films at subject to tensile loading at different strain rates

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### REFERENCES


