Mechanics of 1-25 nm Thin Films

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Abstract:

Nanoscale thin films are used in a wide array of technologies, ranging from computer processing to solar panels. The purpose of this project was to fabricate a device capable of testing mechanical, electrical, and thermal properties of a wide range of free standing thin films within a transmission electron microscope (TEM) environment. Using various photolithography, deposition, and etching techniques, devices were fabricated with free standing platinum thin films. *In situ* TEM experiments on high current-density electromigration in 100 nm platinum thin films were conducted. The specimen began with very small, randomly oriented grains. Current was slowly increased from 100 μA to 10 mA over the course of three and a half hours. Almost instantaneous grain growth was observed at 10 mA. After passing this current for several minutes, average grain size increased dramatically and dislocation pile-up was observed. Selected area electron diffraction (SAED) patterns show grain rotation to the <111> plane during the grain growth. Using the methods and devices developed in this project, a vast array of experiments on nanoscale thin films can be conducted.

Introduction:

It is important to be able to test the mechanical, electrical, and thermal properties of nanoscale thin films, as they often behave significantly different from their bulk material counterparts. The goal of this project was to create a process for fabricating devices for *in situ* transmission electron microscope (TEM) testing of a wide variety of nanoscale thin films. TEM is able to give qualitative information on mechanics, which, coupled with quantitative data from the device, gives great detail on the behavior of these thin films.

Experimental Procedure:

The device design, shown in Figure 1, incorporated mechanical, electrical, and thermal testing. Area 1 of the figure shows the thermal actuators, which expanded due to Joule heating when current was passed through them, imparting stress on the specimen. Area 2 shows the cooling fins which limited thermal leakage to the specimen. Area 3 shows the microelectrodes which allowed for electrical readings using a four-probe method. Thermal information could also be gathered from these microelectrodes using the 3ω method. Area 4 is the thin film itself, made free-standing by the TEM through-hole.

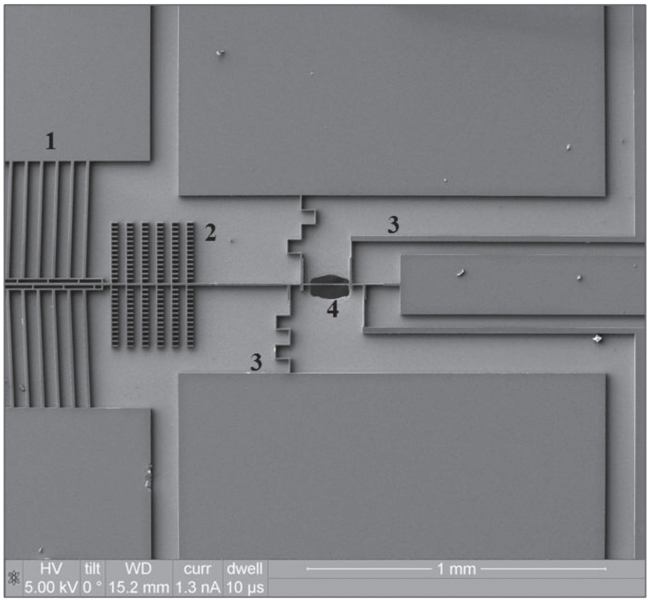

Figure 1: Scanning electron microscope image of device design.

Evaporation deposition techniques were used to deposit 100 nm platinum on the device side of a silicon-on-insulator wafer. The device was fabricated using device and back side photolithography patterning followed by deep reactive ion etching on both sides. Finally, the device was released using hydrofluoric vapor etching.

An electromigration experiment was performed on the platinum sample within the TEM. Current was passed through the sample and slowly increased from 100 μA to 10 mA over three and a half hours.

Results:

Initial TEM, shown in Figure 2, indicated that the film began with a very small average grain size, approximately 5 nm. Initial SAED patterns, seen in the upper right of Figure 2, indicated that the sample had a randomly oriented face centered cubic crystalline structure, similar to that of a powder.

After passing 10 mA current for approximately one minute, the size of the grains increased significantly. SAED indicated that the sample maintained a random crystal structure throughout this growth. The 10 mA current was passed through the sample again and more grain growth was observed. Once the grains reached a certain size, approximately 50-100 nm, dislocation pile-up was seen in TEM imaging, as shown in Figure 3. Most grains ceased to rotate upon reaching this size, indicating that the primary mechanism for accommodating stress had shifted from grain rotation to dislocation motion.

Current was increased to 13 mA, after which the specimen failed. TEM imaging of the remaining areas showed an average grain size of approximately 100 nm, as seen in Figure 4. In some areas of the remaining sample, both the <111> and <200> rings in the SAED pattern vanished completely, while the <220> ring was brightly illuminated, as seen in the upper-right of Figure 4, indicating the grains had almost completely rotated such that the <111> plane was in alignment with the stress direction.

Conclusions:

Significant amounts of grain growth resulted from the high density current passing through the nanoscale platinum thin film, changing the average grain size from ~ 5 nm to ~ 100 nm. The observed grain growth is most likely due to atomic diffusion as a result of electromigration. However, further studies are needed to precisely determine the underlying cause for this grain growth. Specifically, additional studies are needed to isolate the effects of electromigration from any thermal effects due to Joule heating from the electric current that may be contributing to the observed phenomena.

Additionally, the primary mechanism for stress accommodation changed from grain growth and grain rotation to dislocation motion when the grains become large enough to accommodate dislocation pile up. Before this point, the grains rotated to orient the <111> plane in alignment with the stress. In face-centered cubic structures, <111> is the closed-packed family of planes. Therefore, when grains are too small to accommodate dislocation pile-ups, they tend to rotate such that the closed-packed plane is in alignment with the stress.

This experiment demonstrated that our method for creating devices for *in situ* transmission electron microscopy testing of nanoscale thin film mechanics was successful and is viable for future experiments on a vast array of thin films. Devices are currently being fabricated with zinc oxide, aluminum, and poly(3,4-ethylenedioxythiophene) thin films.

Acknowledgments:

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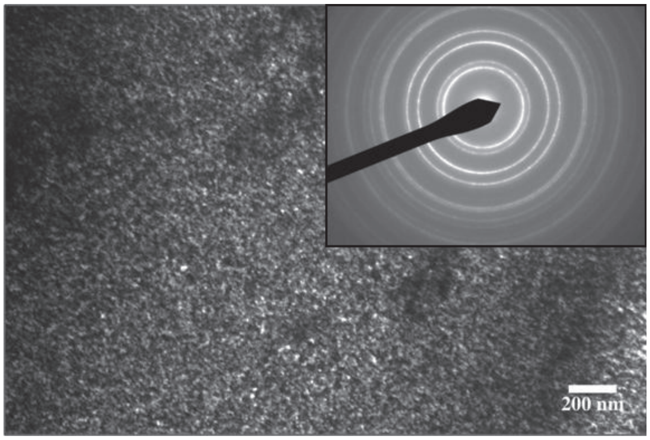

Figure 2: Initial very small grains with random crystalline structure.

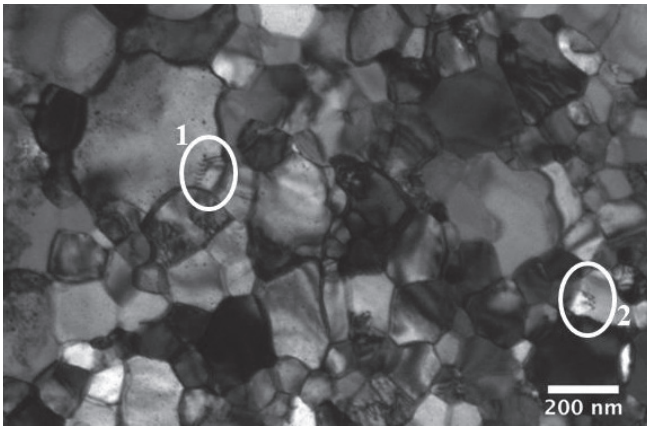

Figure 3: Dislocation pile-up observed in larger grains.

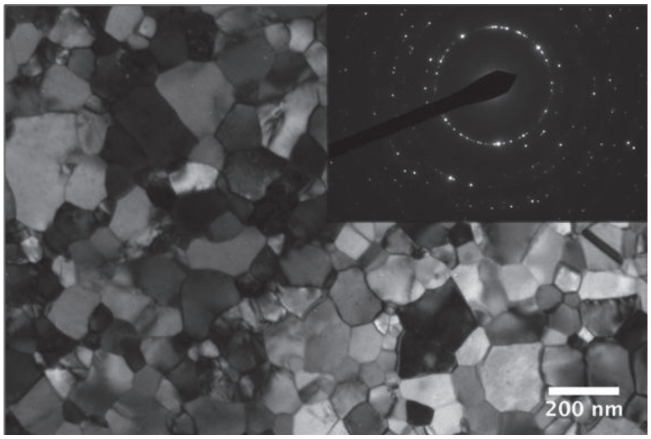

Figure 4: 100 nm grain size with oriented crystalline structure.