Electrical and Structural Properties of Polycrystalline and Epitaxial TiN Films Grown by Reactive Magnetron Sputtering

Friðrik Magnus¹, Árni S. Ingason¹, Sveinn Ólafsson¹ and Jón Tómas Guðmundsson¹,²

¹Science Institute, University of Iceland, Reykjavik, Iceland
²UM-SJTU Joint Institute, Shanghai Jiao Tong University, Shanghai, China

tumi@hi.is

2011 MRS Spring Meeting
San Francisco, California, April 28. 2011
Ultrathin conducting films are an essential part of modern microelectronics.

Titanium nitride (TiN) thin films are widely used in microelectronics:
- as adhesion layers
- as diffusion barriers in device interconnects
- as a direct-metal-gate material for metal-oxide-semiconductor devices

With device dimensions constantly shrinking, the required film thickness is approaching a few nanometers.

For such thicknesses the continuity of a metallic film becomes an important issue.
Ultrathin TiN films were grown by reactive magnetron sputtering on amorphous SiO\textsubscript{2} substrates and single-crystalline MgO substrates at various growth temperatures. The resistance of the films was monitored in-situ during growth to determine the coalescence and continuity thicknesses.

- Growth of ultra-thin TiN films on SiO\textsubscript{2}
  - Coalescence thickness and continuity thickness
  - Structural properties
  - Comparison to films grown by HiPIMS

- Growth of ultra-thin TiN films on MgO
  - Coalescence thickness and continuity thickness
  - Structural properties

- Summary
Growth of ultra-thin TiN films on SiO$_2$
Ultrathin TiN films were grown by reactive dc magnetron sputtering on thermally oxidized Si (100) substrates. The film electrical resistance was monitored *in-situ* during growth in order to determine the minimum thickness of a continuous film. The film texture was examined *ex-situ* by grazing incidence X-ray diffraction (GI-XRD) measurements.
Growth of ultra-thin TiN films on SiO$_2$

- The TiN thin films were grown in a custom built magnetron sputtering chamber.
- The differential resistance of the TiN film was measured in a standard fourpoint probe configuration during growth using dual lock-in amplifier setup.

Growth of ultra-thin TiN films on SiO$_2$

- The nominal coalescence thickness was determined by finding the maximum of $Rd^2$ vs. the film nominal thickness $d$
- The nominal film thickness which completely covers the substrate was determined by the minimum of $Rd^2$ vs. $d$
- $R$ is the in-situ measured film resistance


Growth of ultra-thin TiN films on SiO$_2$

- The film thickness at which the film coalesces (circles) and the film completely covers the substrate (squares), as a function of growth temperature.
- The continuity thickness decreases with increased growth temperature.
- This can be attributed to the increased mobility of the Ti(N) on the surface of the TiN islands with temperature, which causes the voids in the film to be filled more efficiently.

Growth of ultra-thin TiN films on SiO$_2$

- GI-XRD measurements of the 40 nm thick TiN films demonstrate that the films are polycrystalline and that the [111], [200] and [220] crystal orientations are all present in samples grown at 600 °C.
- The [200] peak increases with increasing growth temperature.
- The cross-over of crystal orientation from [200] to [111] is only expected to start occurring at a thickness of 20–50 nm.

Growth of ultra-thin TiN films on SiO$_2$ by HiPIMS
Growth of ultra-thin TiN films on SiO$_2$ by HiPIMS

- TiN thin films were also grown on SiO$_2$ by reactive high power impulse magnetron sputtering (HiPIMS)
- The size of the [111]-peak in low temperature grown HiPIMS suggests that the HiPIMS process encourages the formation of crystallites at low temperature
  

- This has also been seen by TEM in TiN thin films grown at ambient temperature by HiPIMS
  
The grain size of the [111] and [200] crystallites versus growth temperature

The [200] crystallites are smaller than the [111] crystallites

The crystallites are smaller for a HiPIMS grown film than for a dcMS grown films (average grain size for $T_g = 600 \, ^\circ C$ is 30 nm)

HiPIMS grown films reach 5 g/cm$^3$ density at lower growth temperature than dcMS or $T_g = 400^\circ$ C.

HiPIMS produces a much smoother surface than dcMS.

The deposition rate is about 30% lower for HiPIMS than for dcMS.

Growth of ultra-thin TiN films on MgO
Growth of ultra-thin TiN films on MgO

- MgO has a NaCl-type crystal structure with a lattice constant of 4.2112 Å
- TiN which has the same crystal structure and a lattice constant of 4.2417 Å
- It is well known that TiN grows epitaxially on single-crystalline MgO(001) at a substrate temperature above 600 °C


Magnus et al., MRS Proc. 1156 D03-05 (2009)
The nominal coalescence thickness was determined from the maximum of $Rd^2$ vs. the film nominal thickness $d$.

The nominal film thickness which completely covers the substrate was determined by the minimum of $Rd^2$ vs. $d$.

Magnus et al. *Thin Solid Films* accepted 2011
Growth of ultra-thin TiN films on MgO

- As the growth temperature is increased from room temperature to 600 °C
  - The coalescence thickness drops from 1.09 nm to 0.08 nm
  - The thickness for a continuous film drops from 5.5 nm to 0.7 nm

Magnus et al. *Thin Solid Films* accepted 2011
Growth of ultra-thin TiN films on MgO

The films grown at room temperature and 100 °C give peaks corresponding to the [111], [200] and [220] crystal orientations and are therefore clearly polycrystalline.

As the growth temperature is increased to 200 °C there is a clear transition from polycrystallinity to a more ordered crystal structure.

High angle $\theta - 2\theta$ scans show well resolved Laue oscillations on both sides of a distinct TiN Bragg peak, for growth temperatures of 200 °C.

The period is related to the size of the crystallite in the [200] direction (the crystal coherence length or the film thickness).

For films grown at 100 °C the TiN [200] peak is visible, but broad
Growth of ultra-thin TiN films on MgO

- The crystal coherence length calculated from the Laue oscillations $⟨D_{\text{Laue}}⟩$
- We also determine the grain size from the broadening of the Bragg peak using the Scherrer formula $⟨D_{\text{Scherrer}}⟩$
- The film thickness was also determined by X-ray reflectometry (XRR)

Magnus et al. Thin Solid Films accepted 2011
The thickness of the films decreases with increasing growth temperature.

The growth time was 30 min in all cases.

The total thickness of the room temperature grown film is 116.4 nm, or approximately three times that of the 600 °C grown film which is 38.6 nm.

The density is very low for the room temperature grown film, or 2.4 g/cm³, less than half of the bulk TiN density of 5.4 g/cm³.
Summary
Summary – Growth on SiO₂

- Ultrathin TiN films grown by DC magnetron sputtering on SiO₂ substrate temperature ranging from room temperature to 650 °C are polycrystalline.
- We find that the coalescence thickness of the TiN films has a minimum of 1 nm at a growth temperature of 400–500 °C.
- The thickness where the film becomes continuous decreases with increasing growth temperature and is 2.2 nm at 650 °C.
- Films grown at 500 °C and above are resistant to oxidation, indicating a high density, and have a low resistivity of 54 µΩ cm.
- HiPIMS grown films have much smoother surface.
Summary – Growth on MgO

- As the growth temperature is increased from room temperature to 600 °C, the coalescence thickness drops from 1.09 nm to 0.08 nm and the thickness for a continuous film drops from 5.5 nm to 0.7 nm.

- A minimum substrate temperature of 200 °C is required for good epitaxy.

- Films with a density approaching the bulk value and with electrical resistivity of 16.6 $\mu\Omega$ cm can be obtained by raising the growth temperature to 600 °C.

- Substrate temperatures of 100 °C and below yield low density, polycrystalline films.
The slides can be downloaded at http://www.raunvis.hi.is/~tumi/nanocircuits.html


