# Properties of TiN thin films grown on SiO<sub>2</sub> by reactive HiPIMS

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#### Outline

- 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 resistivity and continuity of a metallic film becomes an important issue

- Ultrathin TiN films were grown by reactive dc magnetron sputtering and HiPIMS on amorphous SiO<sub>2</sub> substrates at various growth temperatures
  - Growth of ultra-thin TiN films on SiO<sub>2</sub> by dcMS and HiPIMS

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- Coalescence thickness and continuity thickness
- Structural properties
- Electrical properties
- Exposure to oxygen
- Summary

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- Ultrathin TiN films were grown by reactive dcMS and HiPIMS 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



- 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

Arnalds et al. Rev. Sci. Instrum., 78 103901 (2007)



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

Burgman et al. Thin Solid Films, 474 341 (2005)

Rycroft and Evans Thin Solid Films, 290-291 283 (1996)



- 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

Ingason et al. Thin Solid Films 517 6731 (2009)



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- 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

Mahieu et al. J. Phys.: Conf. Ser., 100 082003 (2008)



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- The room temperature resistivity ρ of 40 nm thick films versus growth temperature, before and after exposure to oxygen
- Exposure to oxygen does not influence the resistivity of films grown at 500 °C and above

Ingason et al. Thin Solid Films 517 6731 (2009)



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- TiN thin films were also grown on SiO<sub>2</sub> 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

Magnus et al. Thin Solid Films 520 1621 (2011)

 This has also been seen by TEM in TiN thin films grown at ambient temperature by HiPIMS

Lattemann et al. Thin Solid Films 518 5978 (2010)



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- 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<sub>g</sub> = 600 °C is 30 nm)

Magnus et al. Thin Solid Films 520 1621 (2011)



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- HiPIMS grown films reach 5 g/cm<sup>3</sup> density at lower growth temperature than dcMS or T<sub>g</sub> = 400° C
- The deposition rate is about 30 % lower for HiPIMS than for dcMS
- HiPIMS produces a much smoother surface than dcMS

Magnus et al. Thin Solid Films 520 1621 (2011)



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- Nominal thickness at which the films coalesce and completely cover the substrate for both HiPIMS and dcMS films
- Both coalescence and continuity thicknesses have a minimum for 400 °C for both growth methods



From Magnus et al. (2012) IEEE EDL 33 1045 (2012)

- The film properties while varying the angle between the target and substrate for T<sub>g</sub> = 400° C
- The density is always higher for HiPIMS grown films
- The growth rate is significanly lower for HiPIMS grown film
- The roughness is much lower for HiPIMS grown film and very uniform



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# Electrical properties of TiN films on SiO<sub>2</sub>

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# Electrical properties of TiN films on SiO<sub>2</sub>

- HiPIMS deposited films have significantly lower resistivity than dcMS deposited films on SiO<sub>2</sub> at all growth temperatures due to reduced grain boundary scattering
- Thus, ultrathin continuous TiN films with superior electrical characteristics can be obtained with HiPIMS at reduced temperatures



From Magnus et al. (2012) IEEE EDL 33 1045 (2012)

# Electrical properties of TiN films on SiO<sub>2</sub>

- Regardless of growth method, resistivity is reduced with increasing growth temperature
- The HiPIMS films are much more resistant to oxidation than the dcMS films
- The change in resistivity for RT grown films is 300 % in dcMS and 17 % in HiPIMS films
- This change can be reduced to 5 % in HiPIMS-deposited films by raising the temperature to 100 ° C



From Magnus et al. (2012) IEEE EDL 33 1045 (2012)

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# The $Ar/N_2$ discharge

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#### HiPIMS - Voltage - Current - time

- The current waveform in the reactive Ar/N<sub>2</sub> HiPIMS discharge is highly dependent on the pulse repetition frequency, unlike for pure Ar
- The current is found to increase significantly as the frequency is lowered
- This is attributed to an increase in the secondary electron emission yield during the self-sputtering phase, when the nitride forms on the target at low frequencies



From Magnus et al. (2011) JAP 110 083306

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### HiPIMS - Voltage - Current - time

- At high frequencies, a nitride is not able to form between pulses, and self-sputtering by Ti<sup>+</sup>-ions (singly and multiply charged) from a Ti target is the dominant process
- At low frequency, the long off-time result in a nitride layer being formed on the target surface and self-sputtering by Ti<sup>+</sup>- and N<sup>+</sup>-ions from TiN takes place
- The observed changes in the discharge current are reflected in the flux of ions impinging on the substrate



From Magnus et al. (2011), JAP 110 083306

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# Summary

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# Summary – Growth on SiO<sub>2</sub>

- Ultrathin TiN films grown on SiO<sub>2</sub> 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 0.5 nm at a growth temperature of 400-500 °C for both dcMS and HiPIMS
- The thickness where the film becomes continuous decreases with increasing growth temperature and is < 2.5 nm for 400-600 °C
- Films grown by HiPIMS have much lower electrical resistivity and are more resistant to oxidation
- HiPIMS grown films have much smoother surface

#### References

#### The slides can be downloaded at

http://langmuir.raunvis.hi.is/~tumi/nanocircuits.html

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