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

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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 continuity of a metallic film becomes an important issue

Outline

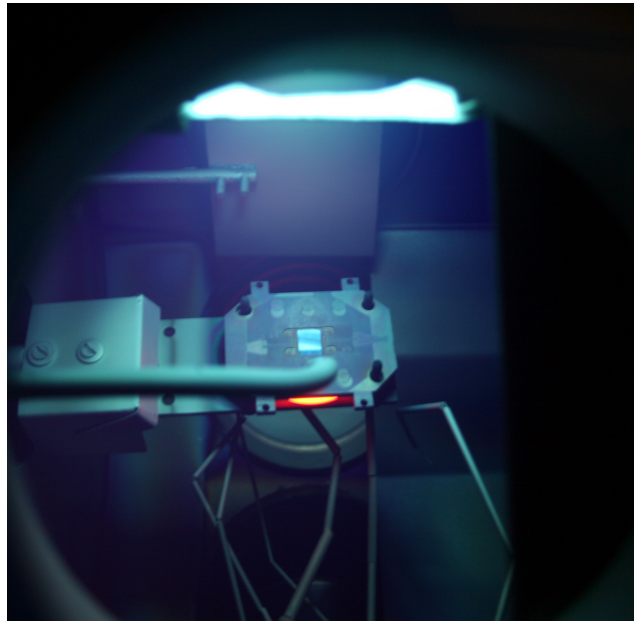
- Ultrathin TiN films were grown by reactive magnetron sputtering on
  - amorphous SiO_2 substrates
  - single-crystalline MgO substrates

  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_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

Growth of ultra-thin TiN films on SiO_2

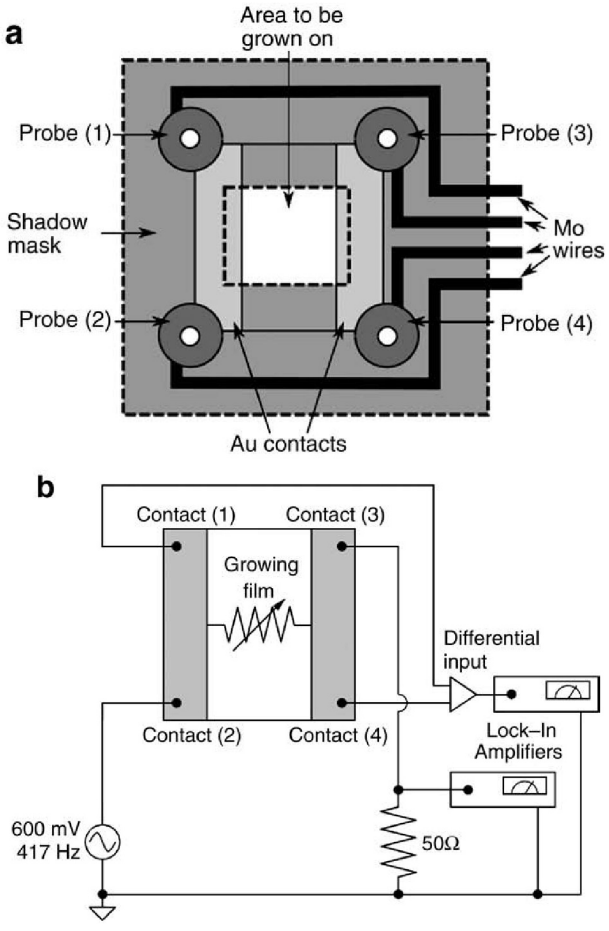
- Ultrathin TiN films were grown by reactive dc magnetron sputtering on thermally oxidized Si (100) substrates
- The film electrical resistance was monitored *in-situ* during growth in order to determine the minimum thickness of a continuous film
- The film texture was examined *ex-situ* by grazing incidence 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

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

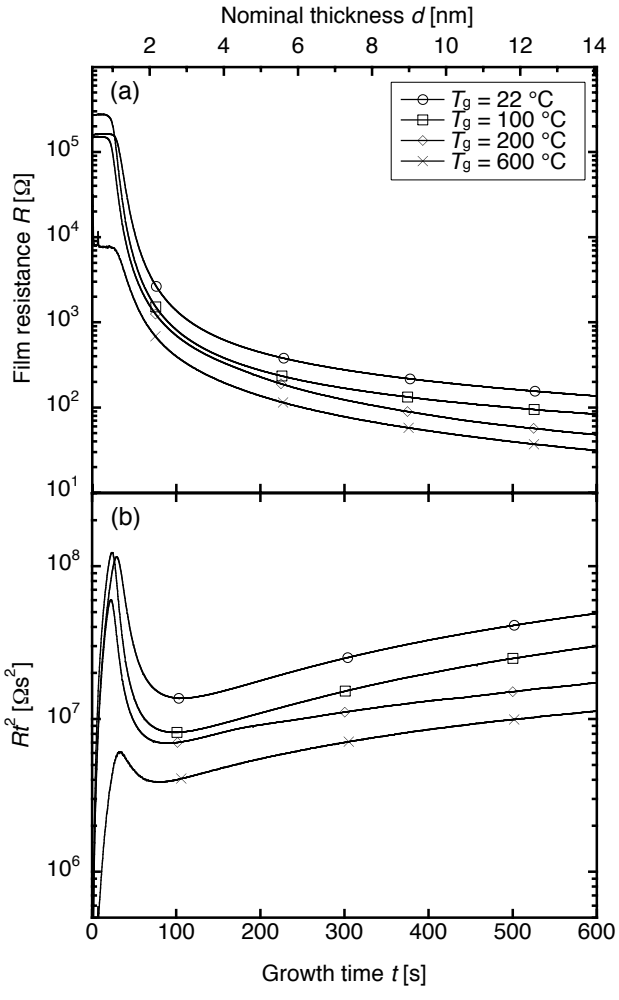


Growth of ultra-thin TiN films on SiO_2

- 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

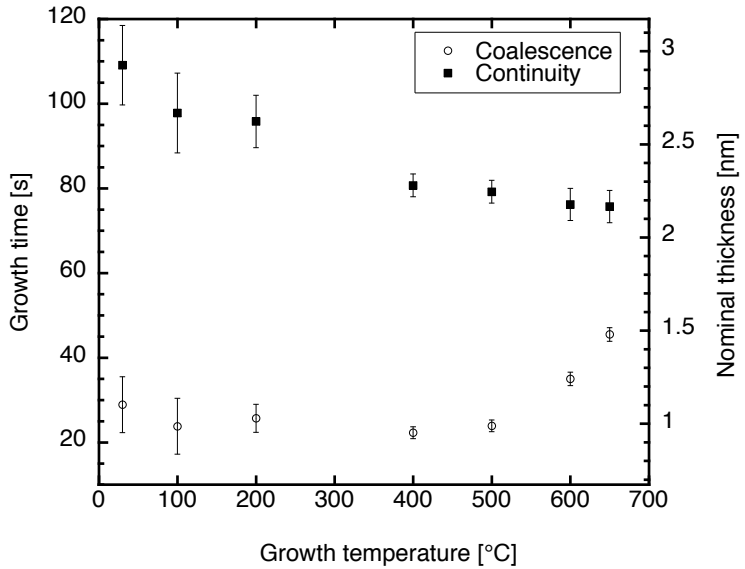
Burgman et al. *Thin Solid Films*, **474** 341 (2005)

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



Growth of ultra-thin TiN films on SiO_2

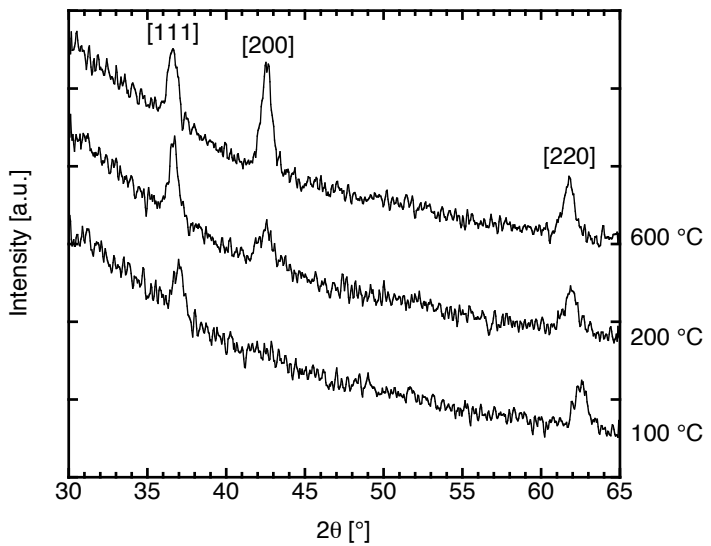
- The film thickness at which the film coalesces (circles) and the film completely covers the substrate (squares), as a function of growth temperature
- The continuity thickness decreases with increased growth temperature
- This can be attributed to the increased mobility of the Ti(N) on the surface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



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

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



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

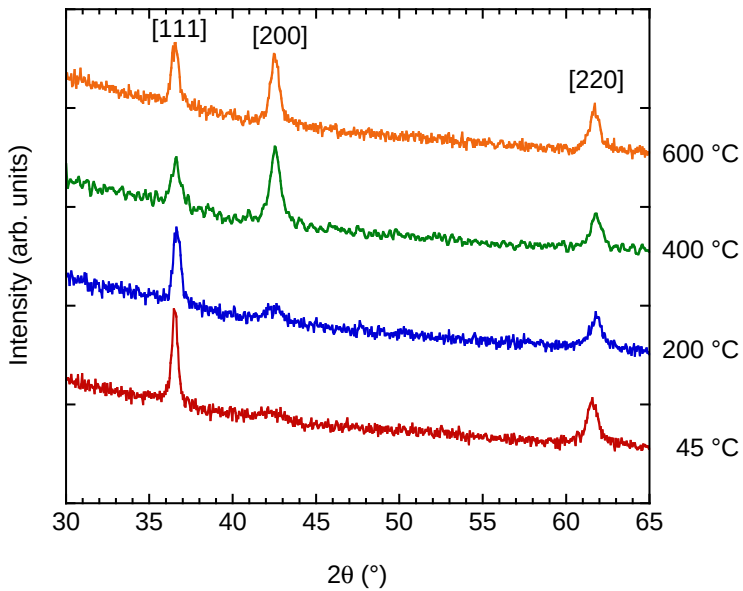
Growth of ultra-thin TiN films on SiO_2 by HiPIMS

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

  Magnus et al. *Thin Solid Films* submitted 2011
- This has also been seen by TEM in TiN thin films grown at ambient temperature by HiPIMS

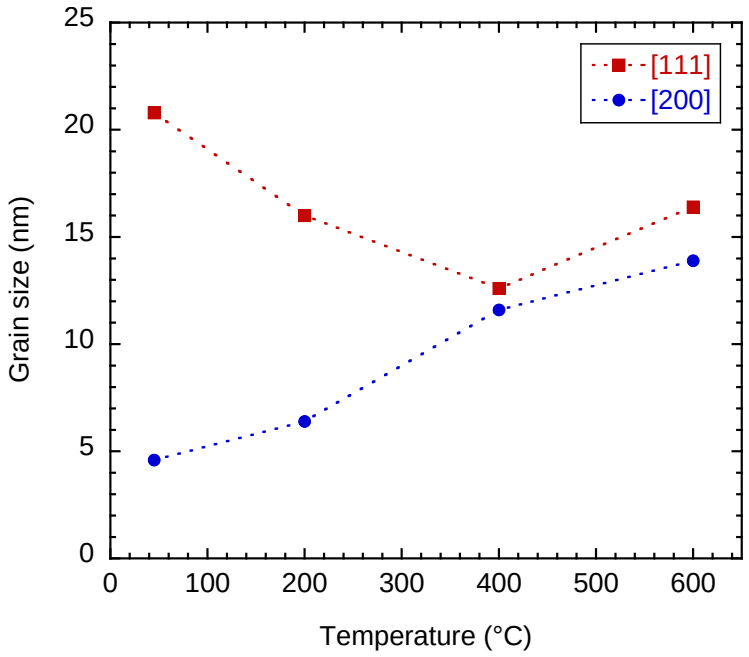
  Lattemann et al. *Thin Solid Films 518* 5978 (2010)



Growth of ultra-thin TiN films on SiO_2 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)

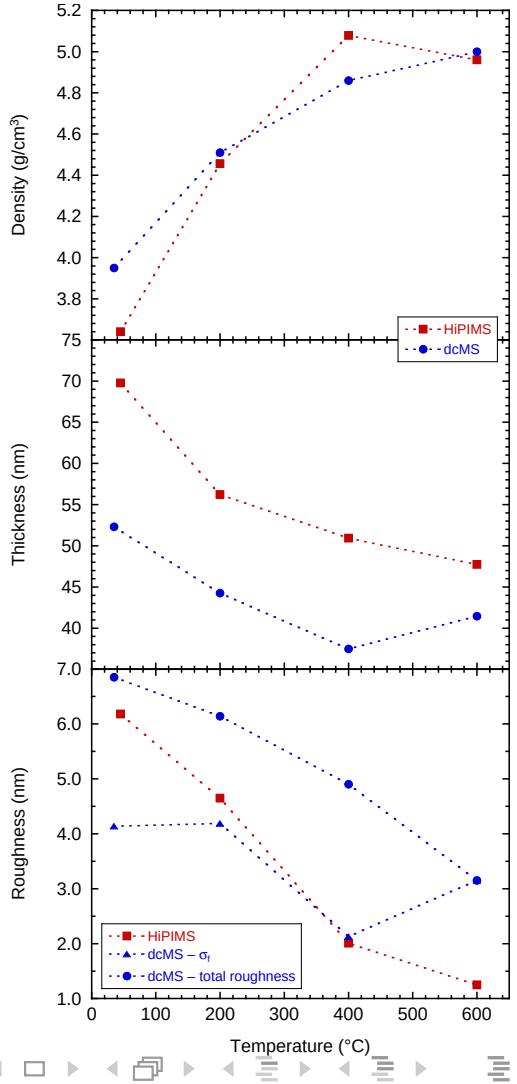
Magnus et al. *Thin Solid Films* submitted 2011



Growth of ultra-thin TiN films on SiO_2 by HiPIMS

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

Magnus et al. *Thin Solid Films* submitted 2011



Growth of ultra-thin TiN films on MgO

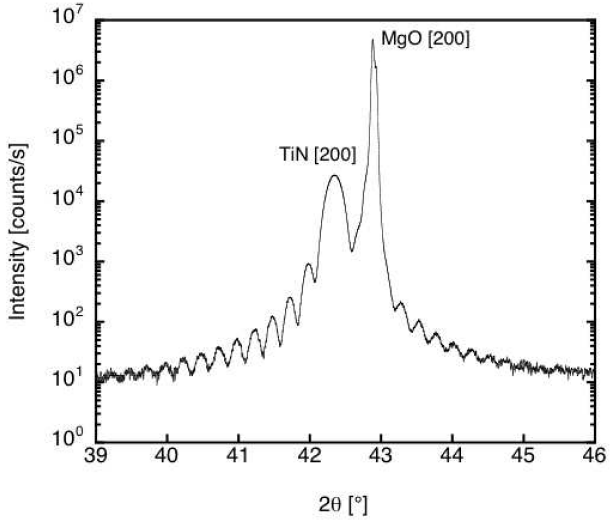
Growth of ultra-thin TiN films on MgO

- 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

Kutschej et al., Thin Solid Films **516** 369 (2007)

Pryds et al., Appl. Phys. A, **93** 705 (2008)

Shin et al., J. Appl. Phys., **95** 356 (2004)

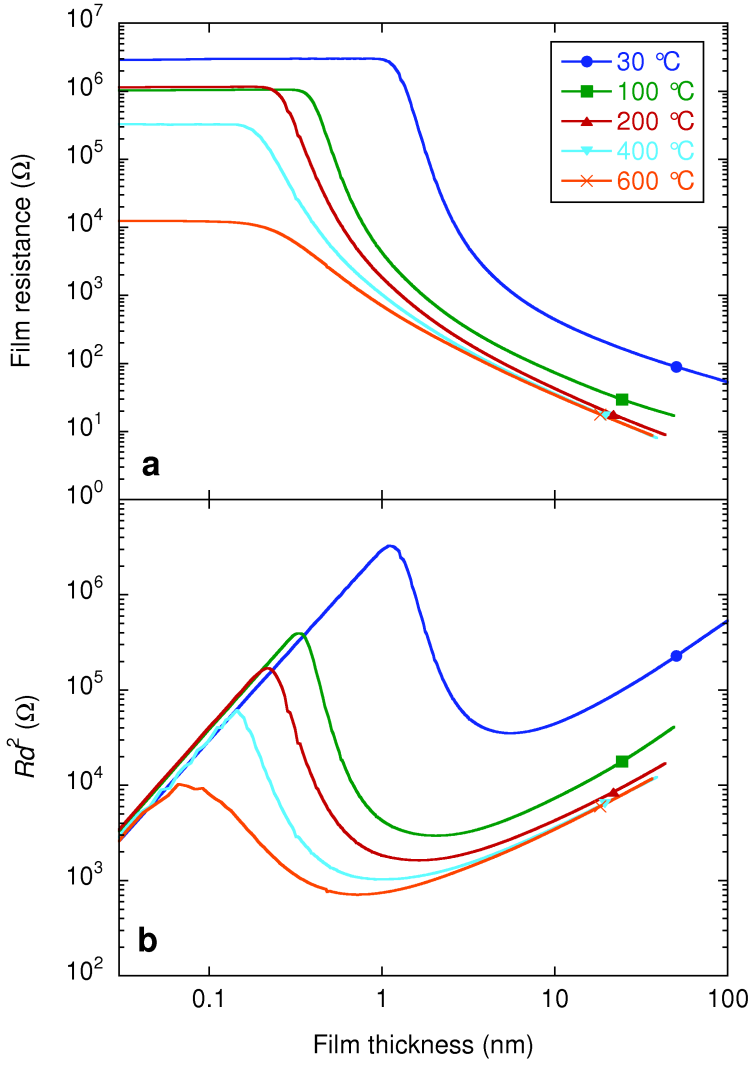


Magnus et al., MRS Proc. **1156** D03-05 (2009)

Growth of ultra-thin TiN films on MgO

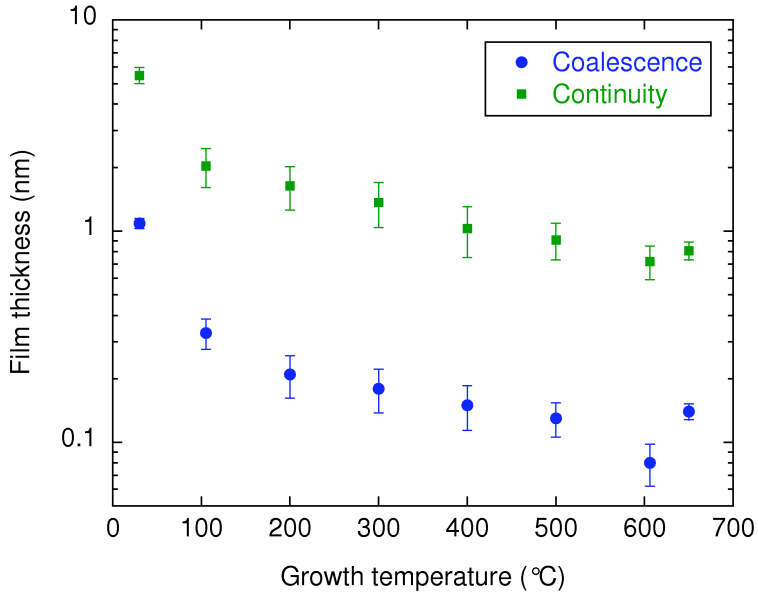
- The nominal coalescence thickness was determined from the maximum of Rd^2 vs. the film nominal thickness d
- The nominal film thickness which completely covers the substrate was determined by the minimum of Rd^2 vs. d

Magnus et al. *Thin Solid Films* accepted 2011



Growth of ultra-thin TiN films on MgO

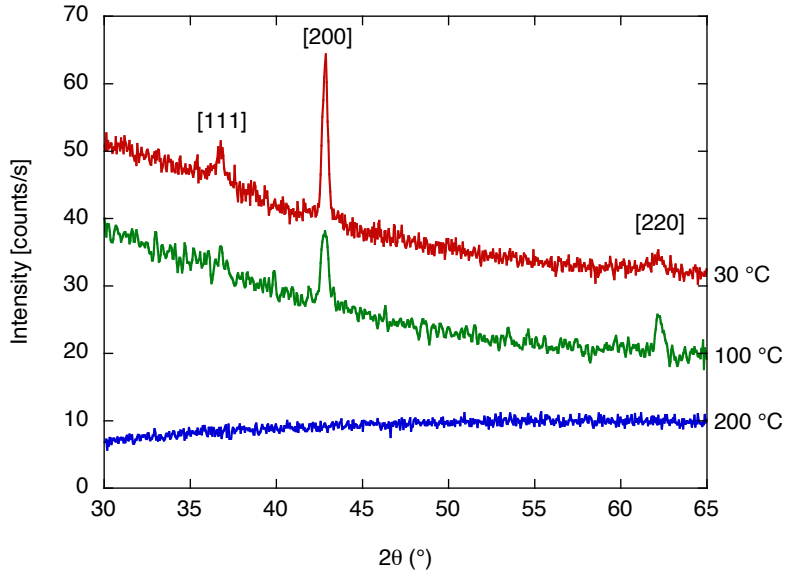
- As the growth temperature is increased from room temperature to 600 °C
  - The coalescence thickness drops from 1.09 nm to 0.08 nm
  - The thickness for a continous film drops from 5.5 nm to 0.7 nm



Magnus et al. *Thin Solid Films* accepted 2011

Growth of ultra-thin TiN films on MgO

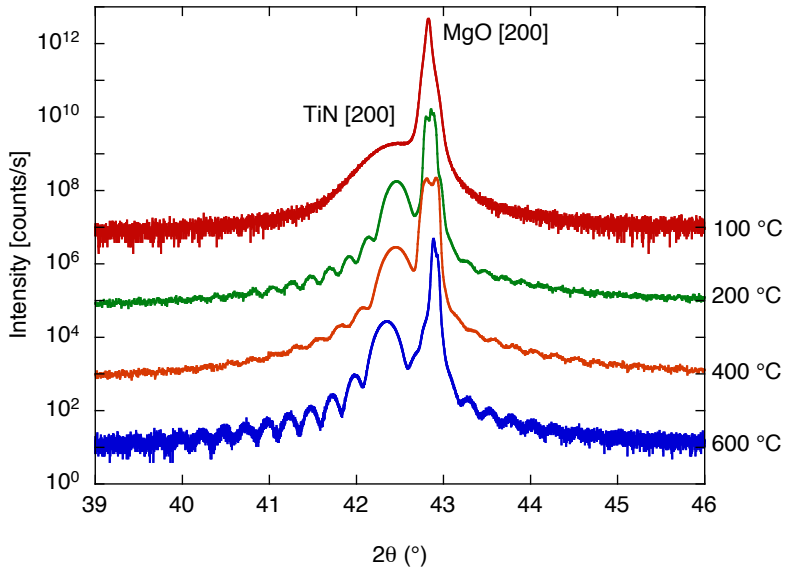
- The films grown at room temperature and 100 °C give peaks corresponding to the [111], [200] and [220] crystal orientations and are therefore clearly polycrystalline
- As the growth temperature is increased to 200 °C there is a clear transition from polycrystallinity to a more ordered crystal structure



Ingason et al. *J. Vac. Sci. Technol. A* **28** 912 (2010)

Growth of ultra-thin TiN films on MgO

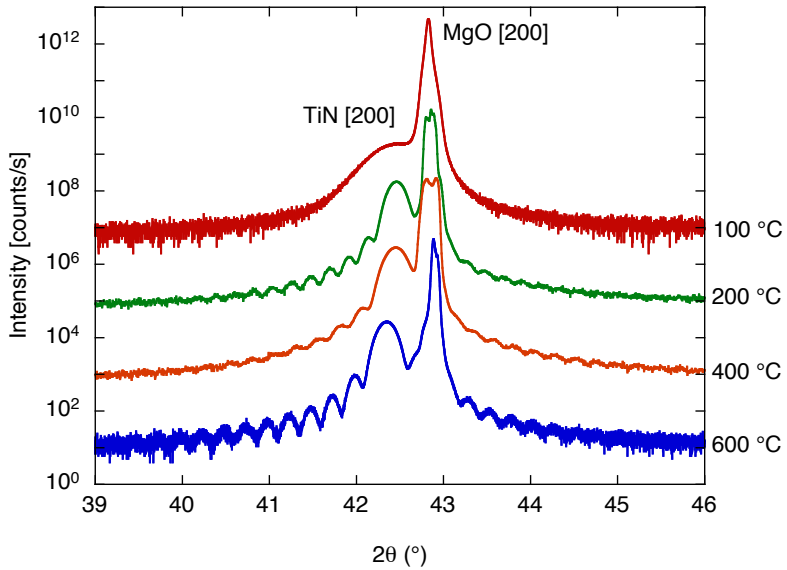
- High angle $\theta - 2\theta$ scans were carried out in order to examine the morphology
- Scans of the room temperature grown films did not reveal a TiN [200] peak indicating that these films are highly polycrystalline
- Even for films grown at 100 °C the TiN [200] peak is visible, although it is quite broad



Ingason et al. *J. Vac. Sci. Technol. A* **28** 912 (2010)

Growth of ultra-thin TiN films on MgO

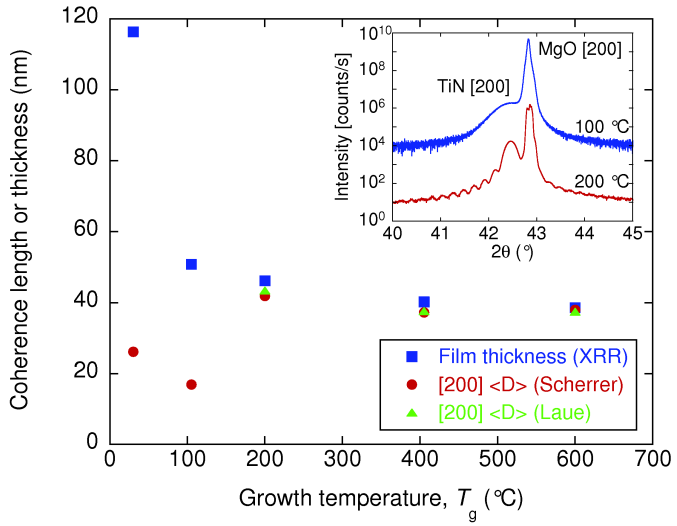
- For growth temperatures of 200 °C and above we see well resolved Laue oscillations on both sides of a distinct TiN Bragg peak
- The period of the Laue oscillations is related to the size of the crystallite in the [200] direction (the crystal coherence length or the film thickness)



Growth of ultra-thin TiN films on MgO

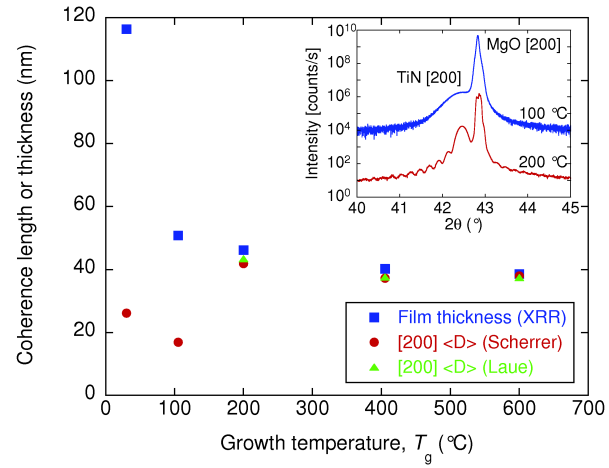
- The crystal coherence length calculated from the Laue oscillations $\langle D_{\mathrm{Laue}} \rangle$
- We also determine the grain size from the broadening of the Bragg peak using the Scherrer formula $\langle D_{\mathrm{Scherrer}} \rangle$
- The film thickness was also determined by X-ray reflectometry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

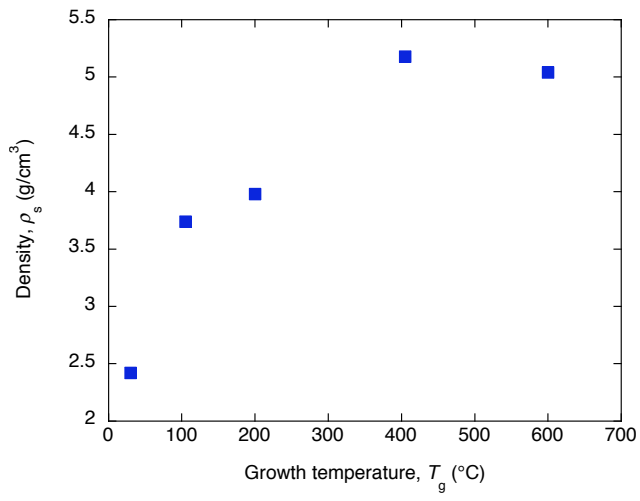
Magnus et al. *Thin Solid Films* accepted 2011



Growth of ultra-thin TiN films on MgO

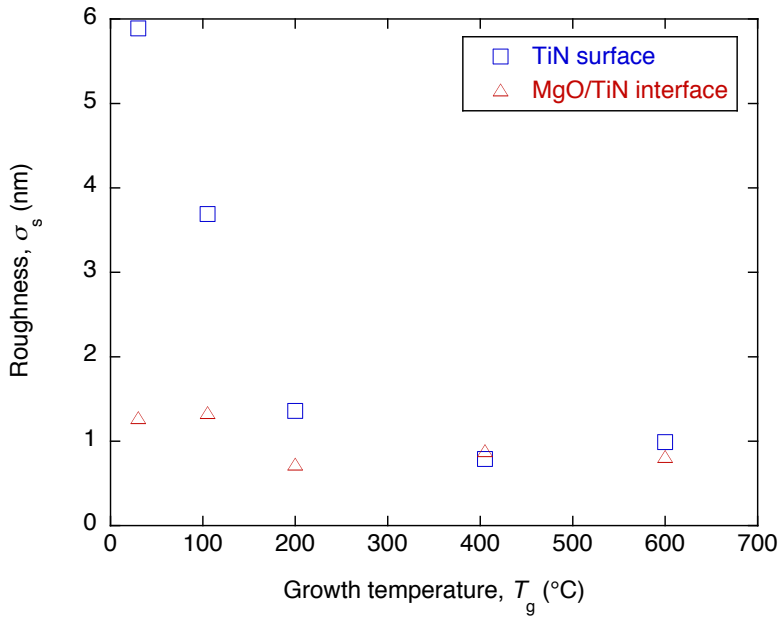
- The thickness of the films decreases with increasing growth temperature
- The growth time was 30 min in all cases
- The total thickness of the room temperature grown film is 116.4 nm, or approximately three times that of the 600 °C grown film which is 38.6 nm
- The density is very low for the room temperature grown film, or 2.4 g/cm^3, less than half of the bulk TiN density of 5.4 g/cm^3





Growth of ultra-thin TiN films on MgO

- The surface and interface roughness was determined by X-ray reflectometry (XRR)
- The TiN/MgO interface roughness is roughly 1 nm in all cases
- The TiN surface roughness is significantly higher for the films grown at low temperature (below 200 °C)



Summary

Summary – Growth on SiO_2

- Ultrathin TiN films grown by DC magnetron sputtering on SiO_2 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 $\mu\Omega\,\mathrm{cm}$
- 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 $\mu\Omega\,\mathrm{cm}$ can be obtained by raising the growth temperature to 600 °C
- Substrate temperatures of 100 °C and below yield low density, polycrystalline films

References

The slides can be downloaded at `http://www.raunvis.hi.is/~tumi/nanocircuits.html`

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