Growth and in-situ electrical characterization of ultrathin epitaxial TiN thin films on MgO

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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 dc 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
  - 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

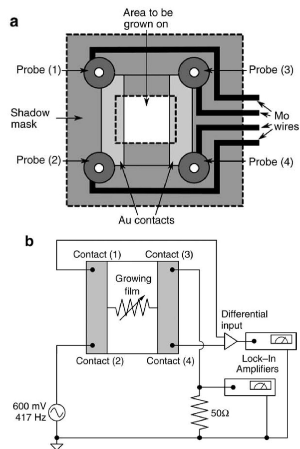
Growth of ultra-thin TiN films on SiO_2

- 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)

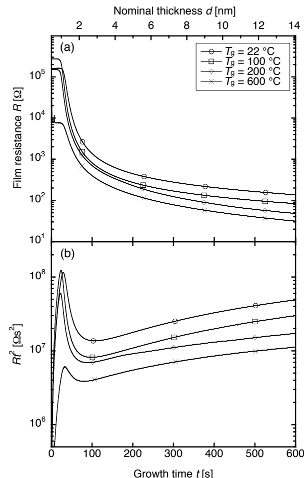


Growth of ultra-thin TiN films on SiO_2

- 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

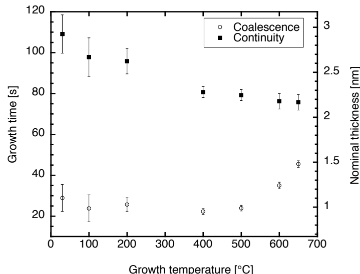
Burgman et al. *Thin Solid Films*, **474** 341 (2005)

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



Growth of ultra-thin TiN films on SiO_2

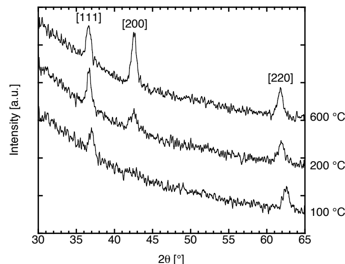
- The deposition time at which the film coalesces (circles) and the film completely covers the substrate (squares), as a function of growth temperature
- The decrease in continuity thickness can be attributed to the increased mobility of the Ti(N) on the surface of the TiN islands with temperature, which 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 MgO

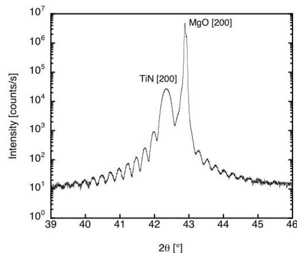
Growth of ultra-thin TiN films on MgO

- 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)

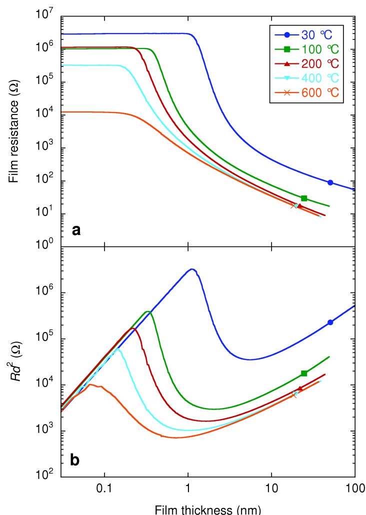


Magnus et al., MRS Proc. **1156** D03-05 (2009)

Growth of ultra-thin TiN films on MgO

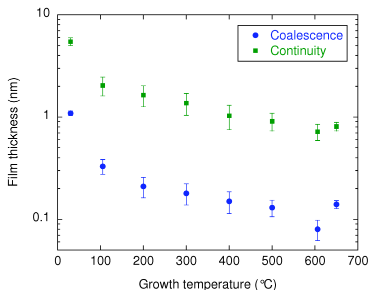
- The nominal coalescence thickness was determined from the maximum of Rd^2 vs. the film nominal thickness d
- The nominal film thickness which completely covers the substrate was determined by the minimum of Rd^2 vs. d

Magnus et al. *Thin Solid Films* submitted 2010



Growth of ultra-thin TiN films on MgO

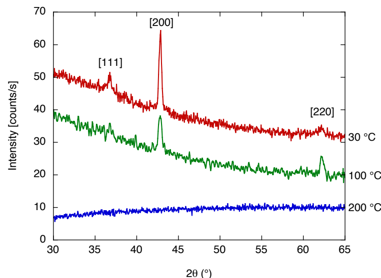
- As the growth temperature is increased from room temperature to 600 °C
  - The coalescence thickness drops from 1.09 nm to 0.08 nm
  - The thickness for a continous film drops from 5.5 nm to 0.7 nm



Magnus et al. *Thin Solid Films* submitted 2010

Growth of ultra-thin TiN films on MgO

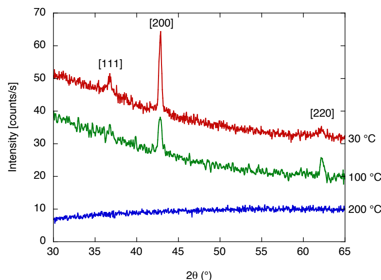
- The films grown at room temperature and 100 °C give peaks corresponding to the [111], [200] and [220] crystal orientations and are therefore clearly polycrystalline
- The scans have been optimized to give the maximum [200] peak intensity and therefore the [111] peak is barely visible in the scan of the 100 °C grown film



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

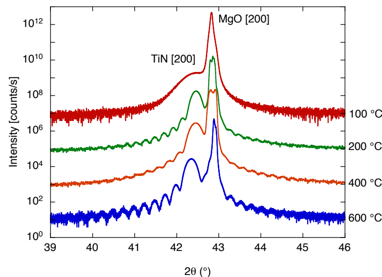
Growth of ultra-thin TiN films on MgO

- As the growth temperature is increased to 200 °C there is a clear transition from polycrystallinity to a more ordered crystal structure
- There are no peaks visible in the GIXRD scan and a significantly reduced background - the lattice planes of this film are parallel with the substrate surface



Growth of ultra-thin TiN films on MgO

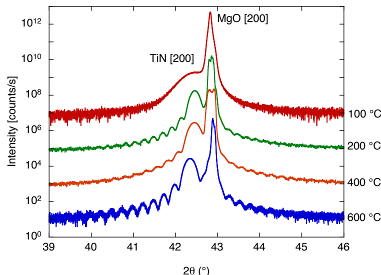
- High angle $\theta - 2\theta$ scans were carried out in order to examine the morphology
- Scans of the room temperature grown films did not reveal a TiN [200] peak indicating that these films are highly polycrystalline
- Even for films grown at 100 °C the TiN [200] peak is visible, although it is quite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



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

Growth of ultra-thin TiN films on MgO

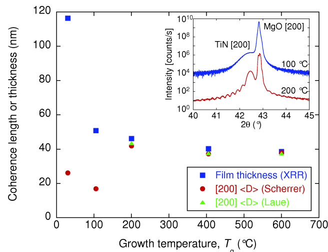
- For growth temperatures of 200 °C and above we see well resolved Laue oscillations on both sides of a distinct TiN Bragg peak
- The period of the Laue oscillations is related to the size of the crystallite in the [200] direction (the crystal coherence length or the film thickness)



Growth of ultra-thin TiN films on MgO

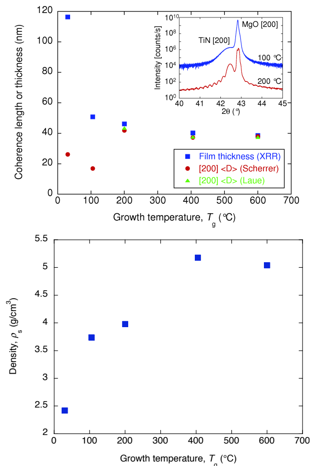
- The crystal coherence length calculated from the Laue oscillations $\langle D_{\text{Laue}} \rangle$
- We also determine the grain size from the broadening of the Bragg peak using the Scherrer formula $\langle D_{\text{Scherrer}} \rangle$
- The film thickness was also determined by X-ray reflectometry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

Magnus et al. *Thin Solid Films* submitted 2010



Growth of ultra-thin TiN films on MgO

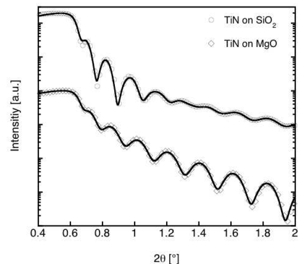
- The thickness of the films decreases with increasing growth temperature
- The growth time was 30 min in all cases
- The total thickness of the room temperature grown film is 116.4 nm, or approximately three times that of the 600 °C grown film which is 38.6 nm
- The density is very low for the room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



Comparison – growth on SiO_2 and MgO at 600°C

Comparison – growth on SiO_2 and MgO at 600°C

- XRR analysis was carried out to determine the film thickness, density and roughness by curve fitting
- The TiN/SiO_2 is best fitted by a two-layer model including a 5.6 nm thick surface oxynitride layer
- The TiN/MgO sample can be fitted with a single TiN layer



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

Substrate	Density (g/cm^3)	Thickness (nm)	Roughness (nm)	Resistivity (μΩ cm)	Coalescence thickness (Å)	Continuity thickness (Å)
SiO_2	4.9	41.7	2.7	54 ± 4	8 ± 1	19 ± 2
MgO	5.1	38.7 (37.5*)	1.0	24 ± 4	2 ± 1	–

* From Laue oscillations

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 $\mu\Omega\,\mathrm{cm}$

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 a crystal coherence length of up to 97 % of the total TiN film thickness can be obtained by raising the 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`

Arnalds, U. B., J. S. Agustsson, A. S. Ingason, A. K. Eriksson, K. B. Gylfason, J. T. Gudmundsson, and S. Olafsson (2007). A magnetron sputtering system for the preparation of patterned thin films and in situ thin film electrical resistance measurements. *Review of Scientific Instruments 78*(10), 103901.

Burgmann, F. A., S. H. N. Lim, D. G. McCulloch, B. K. Gan, K. E. Davies, D. R. McKenzie, and M. M. M. Bilek (2005). Electrical conductivity as a measure of the continuity of titanium and vanadium thin films. *Thin Solid Films 474*(1-2), 341–345.

Ingason, A. S., F. Magnus, J. S. Agustsson, S. Olafsson, and J. T. Gudmundsson (2009). In-situ electrical characterization of ultrathin TiN films grown by reactive dc magnetron sputtering on SiO_2. *Thin Solid Films 517*(24), 6731–6736.

Ingason, A. S., F. Magnus, S. Olafsson, and J. T. Gudmundsson (2010). Morphology of TiN thin films grown on MgO [100] by reactive dc magnetron sputtering. *Journal of Vacuum Science and Technology A 28*(4), 912–915.

Kutschej, K., B. Rashkova, J. Shen, D. Edwards, C. Mitterer, and G. Dehm (2007). Experimental studies on epitaxially grown TiN and VN films. *Thin Solid Films 516*(2-4), 369 – 373.

Magnus, F., A. S. Ingason, S. Olafsson, and J. T. Gudmundsson (2009). Electrical and structural properties of ultrathin polycrystalline and epitaxial TiN films grown by reactive dc magnetron sputtering. In M. Gall, A. Grill, F. Iacopi, J. Koike, and T. Usui (Eds.), *Materials, Processes and Reliability for Advanced Interconnects for Micro- and Nanoelectronics - 2009*, pp. 1156–D03–05. Mater. Res. Soc. Symp. Proc.

Magnus F., A. S. Ingason, S. Olafsson, and J. T. Gudmundsson (2010). Growth and in-situ electrical characterization of ultrathin epitaxial TiN films on MgO_2. *Thin Solid Films* submitted June 2010.

Mahieu, S., D. Depla, and R. De Gryse (2008). Modelling the growth of transition metal nitrides. *Journal of Physics: Conference Series 100*, 082003.

Pryds, N., D. Cockburn, K. Rodrigo, I. L. Rasmussen, J. Knudsen, and J. Schou (2008). Growth of thin films of TiN on MgO(100) monitored by high-pressure RHEED. *Applied physics A: Materials Science & Processing 93*(3), 705–710.

Rycroft, I. M. and B. L. Evans (1996). The in situ characterization of metal film resistance during deposition. *Thin Solid Films 290-291*, 283–288.

Shin, C.-S., S. Rudenja, D. Gall, N. Hellgren, T.-Y. Lee, I. Petrov, and J. E. Greene (2004). Growth, surface morphology, and electrical resistivity of fully strained substoichiometric epitaxial TiN_x ($0.67 <= x < 1.0$) layers on MgO(001). *Journal of Applied Physics 95*(1), 356–362.