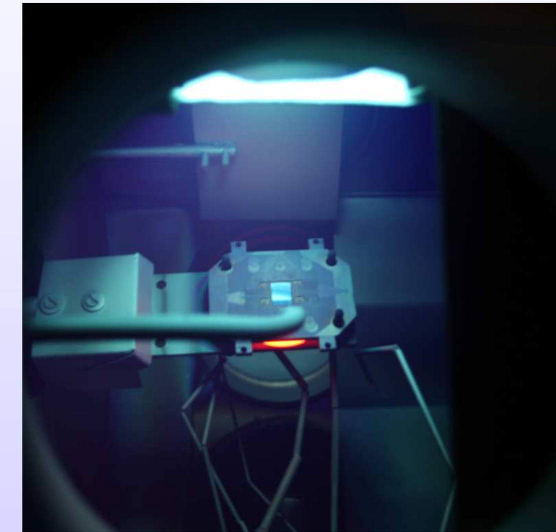
In-situ electrical characterization of ultrathin TiN films grown by reactive dc magnetron sputtering on SiO_2



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Introduction

- Ultrathin TiN films were grown by reactive dc magnetron sputtering on thermally oxidized Si (100) substrates.
- The electrical resistance of the films 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 and the film composition was determined by X-ray photoelectron spectroscopy (XPS).

Experimental apparatus and method

- The TiN thin films were grown in a custom built magnetron sputtering chamber (Arnalds et al., 2007).
- The argon flow rate was 40 sccm and the nitrogen flow rate 2 sccm to give a pressure of 0.4 Pa. The applied power was 100 W.
- The substrates used were thermally oxidized Si(001) with an oxide thickness of 500 nm. Au contact pads were defined using a photolithographic lift-off process prior to TiN deposition.
- The differential resistance of the TiN film was measured in a four-terminal configuration during growth using a dual lock-in amplifier (Arnalds et al., 2007).
- X-ray measurements were carried out using a Philips X'pert diffractometer (Cu K_α, wavelength 0.15406 nm) mounted with a hybrid monochromator/mirror on the incident side and a 0.27° collimator on the diffracted side.

Results and discussion

- The nominal coalescence thickness was determined by finding the maximum of Rd^2 vs. the film nominal thickness d, where R is the *in-situ* measured film resistance (or equivalently Rt^2 vs. growth time t).
- The nominal film thickness which completely covers the substrate was determined by the minimum of Rd^2 vs. d (Burgmann et al., 2005; Rycroft and Evans, 1996).

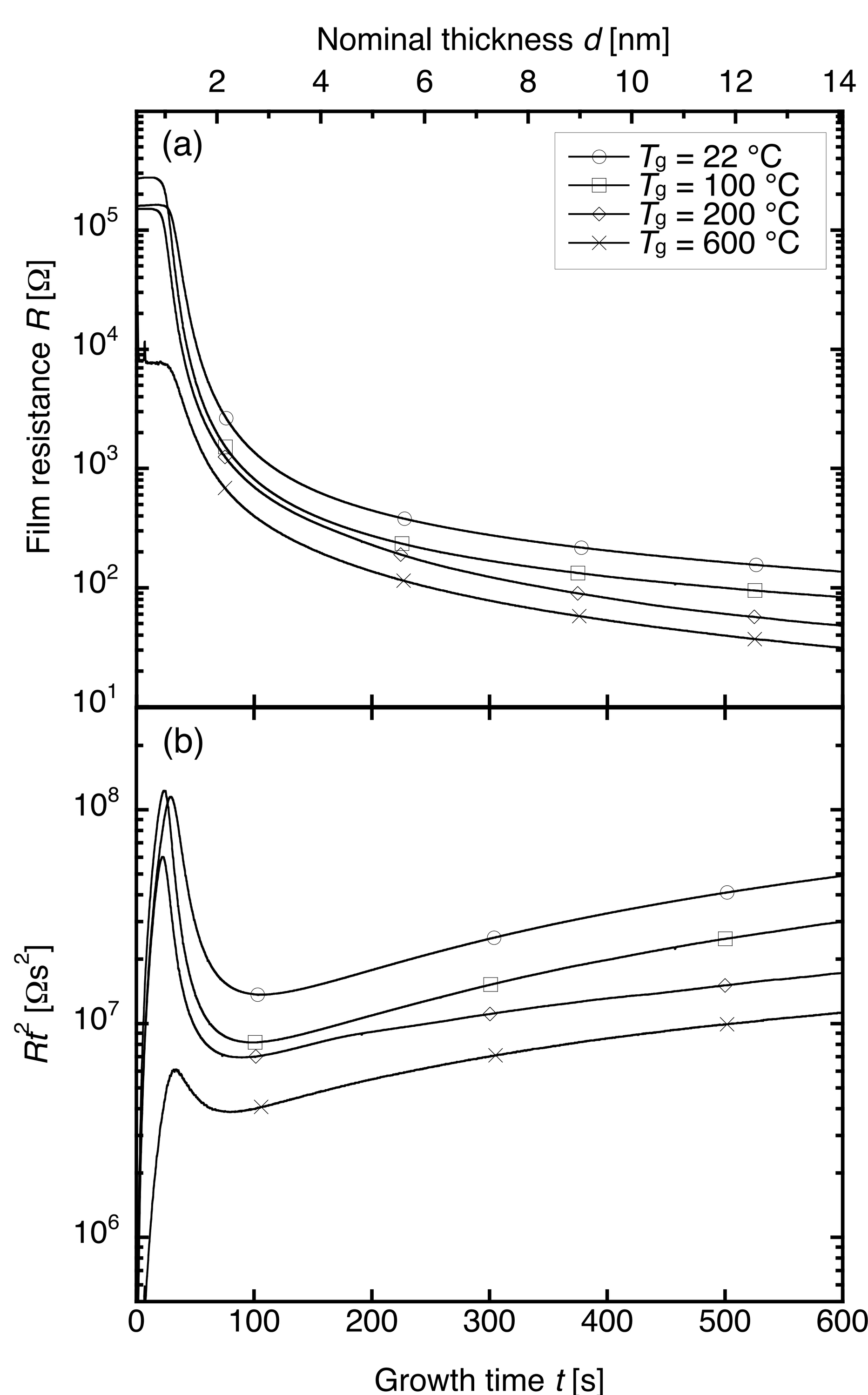

Figure 1: (a) The film resistance R as a function of TiN deposition time t measured *in-situ* during growth, at four different growth temperatures T_g. (b) Rt^2 versus growth time t and nominal film thickness d based on a constant growth rate.

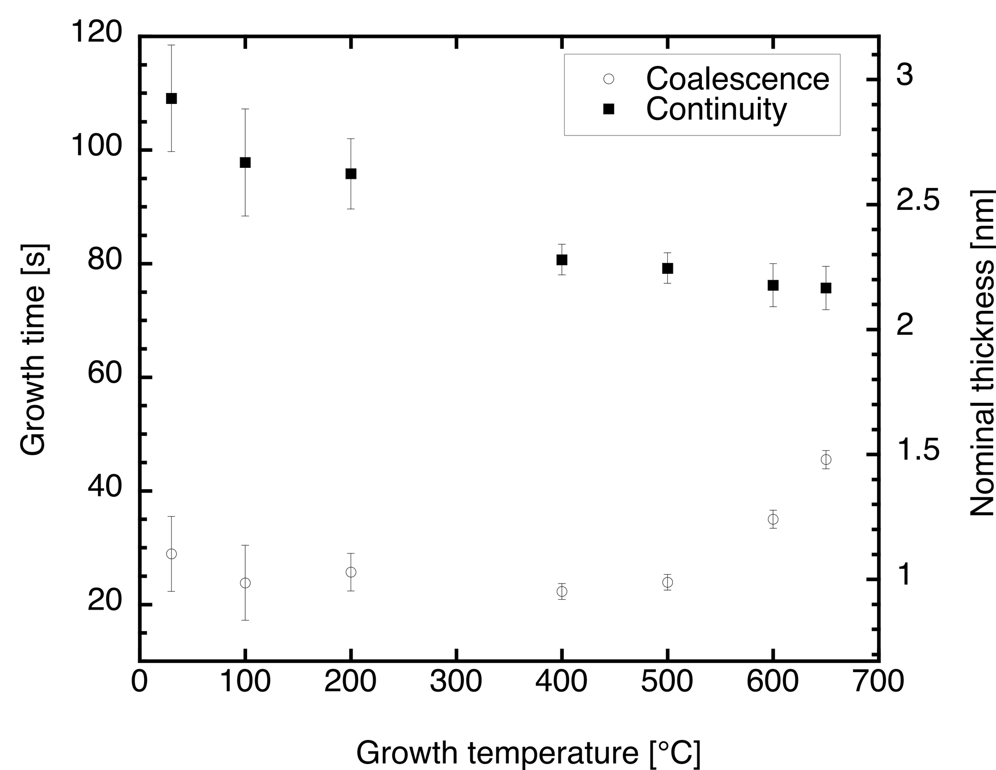

Figure 2: The deposition time at which the film coalesces (circles) and the film completely covers the substrate (squares), as a function of growth temperature. The error bars reflect the spread in growth time over a number of samples. The nominal film thickness based on a constant growth rate is also shown.

- The coalescence thickness is approximately constant below 400 °C, after which it increases sharply with increasing temperature.
- The continuity thickness is seen to decrease with increasing 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.

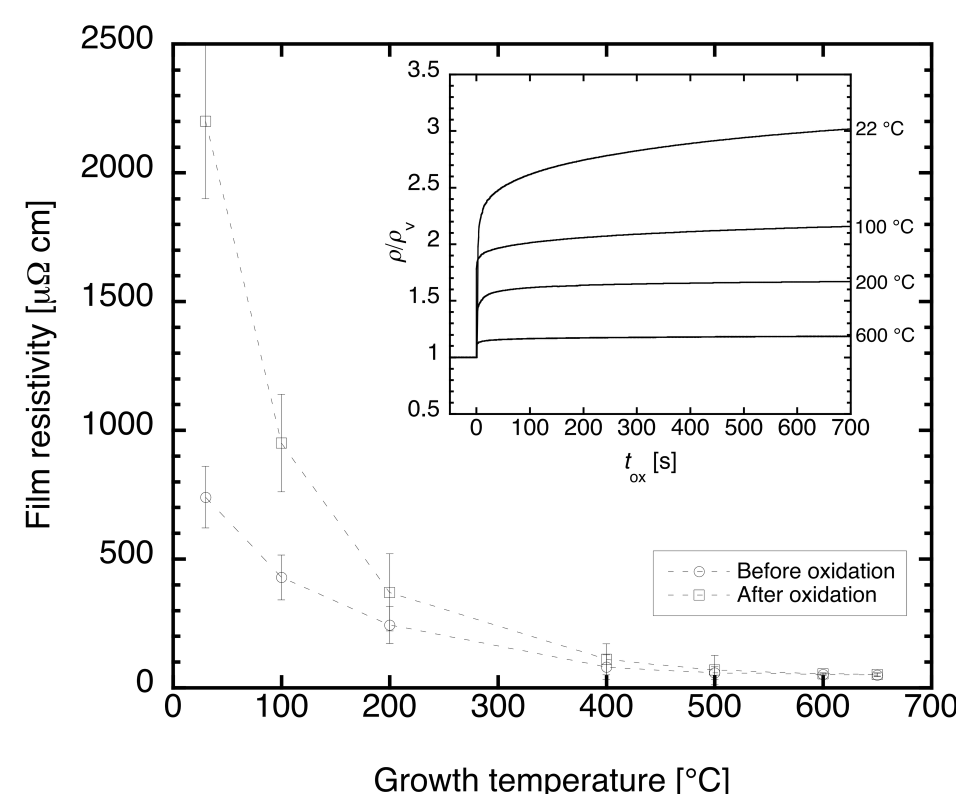

Figure 3: Inset: The evolution of the film resistivity ρ as the film is exposed to oxygen at room temperature. The resistivity is normalized by the resistivity before oxidation, ρ_v. Main graph: Room temperature resistivity ρ of 40 nm thick films versus growth temperature, before and after oxidation.

- After the films had cooled down to room temperature they were exposed to oxygen and the film resistance monitored.
- The resistivity of the room temperature grown films increases by a factor of three as they are exposed to oxygen for 30 minutes, suggesting a highly porous film. In contrast, exposure to oxygen has little effect on the resistivity of films grown at 500 °C and above.
- The XPS measurements reveal that the oxygen content is highest for the films grown at room temperature (∼23%) but decreases with elevated growth temperature, reaching ∼10% for films grown at 600 °C.

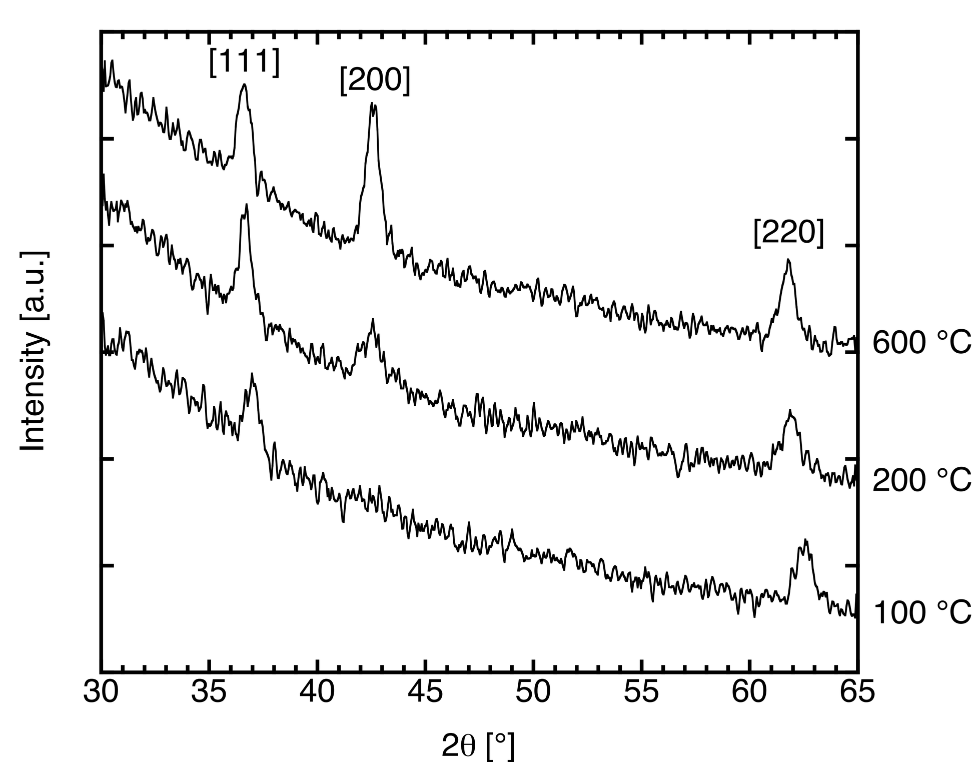

Figure 4: GI-XRD scans of the 40 nm thick TiN films, grown at different temperatures shown on the right. The curves are shifted for clarity.

- The cross-over of crystal orientation from [200] to [111] is only expected to start occurring at a thickness of 20−50 nm so the presence of both orientations is unsurprising.
- These observations are consistent with previous studies and can be attributed to the competition between the temperature dependent fast lateral growth rate of the [200] oriented grains and the large geometric growth rate of the [111] oriented grains (Mahieu et al., 2008).

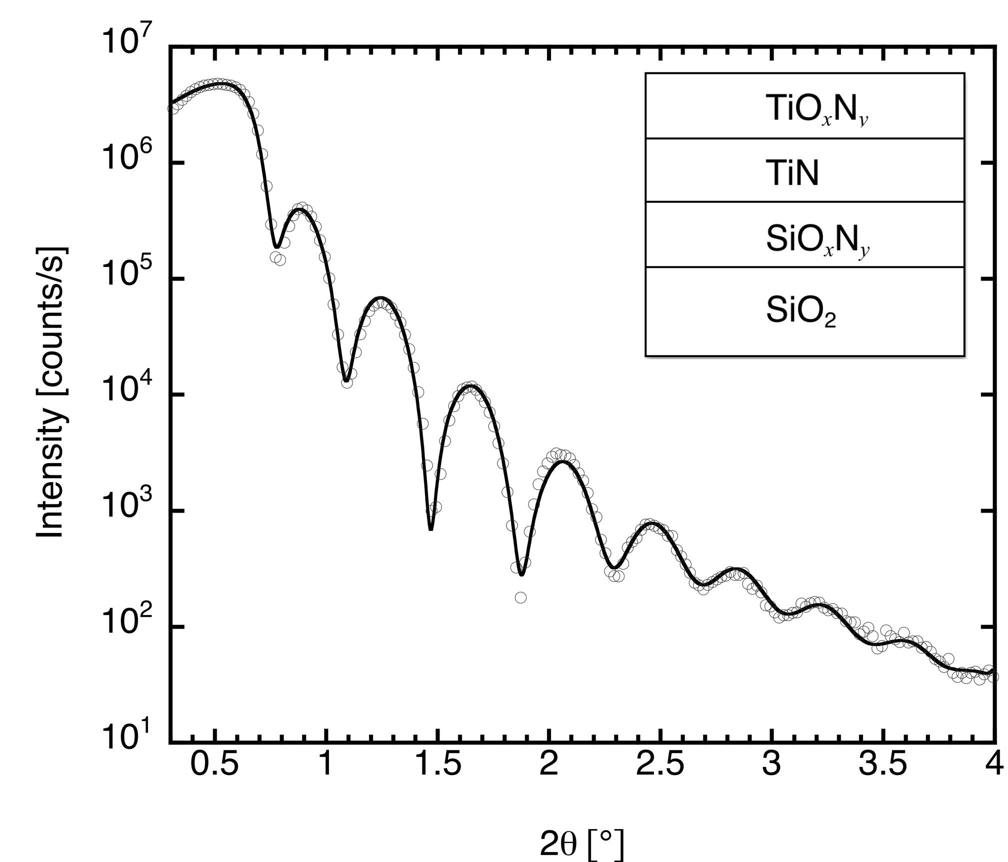

Figure 5: An example of an XRR measurement of a 20 nm thick TiN film (circles) including a fit based on the Parratt formalism for reflectivity (Parratt, 1954) (line). The inset shows the layer model used (not to scale).

- XRR measurements were performed to determine the film thickness, density and roughness.
- The TiN layer density in the 600 °C grown films was found to be $4.9 \pm 0.2\,\mathrm{g/cm^3}$
- In order to simulate the XRR data it was necessary to introduce a ∼2 nm thick layer on top of the TiN layer with a slightly lower density than TiN. This can be attributed to a surface oxynitride layer (Ernsberger et al., 1986).
- In addition, the model included a reaction layer between the TiN and SiO_2 with a density slightly below that of SiO_2.
- The roughness of the top surface (the titanium oxynitride layer) is found to be approximately 1.1 nm and the roughness of the TiN/TiO_xN_y interface is 0.75 nm.

Conclusions

- Ultrathin TiN films have been grown by DC magnetron sputtering with the SiO_2 substrate temperature ranging from room temperature to 650 °C.
- We find that the coalescence thickness of the TiN films has a minimum of 1 nm at a growth temperature of 400−500 °C whereas 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 $\mu\Omega$cm.

Acknowledgments

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