



ROBL-CRG

Experiment title:*In situ* x-ray diffraction during sputter deposition of TiN and CrN**Experiment number:**
20_02_041**Beamline:**

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Report: (A)

The **heteroepitaxial growth of TiN on MgO(001)**, deposited by **reactive magnetron sputtering**, has been studied *in situ*. Using real-time specular X-ray reflectivity, layer-by-layer growth was observed, with the surface-roughening decreasing with increasing deposition temperature. Higher temperatures also resulted in lower growth rates. The film thickness was measured with specular X-ray reflectivity. Using off-plane Bragg-Brentano as well as grazing incidence in-plane wide angle scattering, the pseudomorphic growth of TiN to the underlying MgO(001) was established. Transmission electron microscopy revealed atomic planes passing through the MgO-TiN boundary, thus confirming the heteroepitaxial growth. To the best of our knowledge, this is the first report [1] on *real-time* measurements of the surface morphology on an atomic scale during the growth of a film deposited by magnetron sputtering.

EXPERIMENTAL

The **deposition chamber** (together with the detailed **scattering geometry** and the quality of the data, like intensity, resolution, background, which can be obtained with the set-up) is described in detail in **Ref. 2**. The **magnetrons**, commercially available from AJA International, are placed at a distance of 100 mm from the substrates and tilted 30 degrees away from the substrate normal. To avoid cross contamination of the two targets, each with a diameter of 1 inch, chimneys are mounted on the magnetrons. Air-pressure-controlled shutters are placed in front of the chimneys. The base pressure was appr. 2×10^{-5} Pa. The **reactive sputter gas** was a mixture of Ar (99.9996%) and N₂ (99.99990%) with the ratio 4:1, at a total gas pressure of 0.3 Pa. Only one magnetron was used at the time. It was run at a dc power of 80 W, resulting in a deposition rate of appr. 0.65 Å/s. The **substrates** were 1.5 x 1.5 cm² MgO(001) single crystals with a thickness of 1 mm. A resistive **heater** was mounted below the substrate so the temperature could be varied from room temperature up to 600 °C. The temperature was measured by use of a thermocouple. A negative **bias voltage** of 30 V was applied to the substrate. Before depositing TiN, the surface of the substrate was **cleaned/annealed** by heating it to 600 °C for 1 h.

The deposition chamber was mounted on the six-circle goniometer in MRH. The incident x-rays were monochromatized to 11.696 keV ($\lambda = 1.060$ Å). To study the growth of TiN films *in situ*, four different scattering geometries were used:

