ROBL-CRG	**Experiment title:** *In-situ* **x-ray diffraction during sputter deposition of $Ti_{1-x}Al_xN$ – Part II**	**Experiment number**: **20_02_608**
Beamline: BM 20	**Date of experiment**: from: 12.11.2003 to: 18.11.2003	**Date of report**: 17.11.2004
Shifts: 18	**Local contact(s)**: Dr. Norbert Schell	*Received at ROBL: 17.11.04*

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Report:

The **growth of** magnetron-sputtered $\mathbf{Ti_{1-x}Al_xN}$ thin films on amorphous substrates has been monitored by ***in-situ*** XRD. The **texture** development of these films shows a **strong dependence on deposition rate**, while **low values of the Al concentration x** and the deposition **temperature play a negligible role**. **Suppresing the ion current** to the substrate by a positive bias voltage can completely **reverse the final texture** even for low deposition rates, showing the importance of ion-substrate interaction, in accordance with [1].

EXPERIMENTAL

The $Ti_{1-x}Al_xN$ films have been grown in the ROBL deposition chamber, the characteristics of which are described in detail elsewhere [2]. As substrates, Si(100) wafers with a 1400 Å amorphous oxide capping layer of size 15 x 15 mm have been used. The base pressure before deposition was appr. 1 x 10^{-4} Pa. Ar and N_2 were applied as sputter gases with a gas flow of 2.82 sccm, leading to a constant working pressure of 0.35 Pa. During the reactive co-sputtering process the Ti and Al targets of 99.999 % purity were run at a constant DC power of 80 W, and 0/5 W, respectively. For a reactive gas mixture of Ar/N_2 = 2/0.6 this resulted in a growth rate of ~1 Å/s, and halved down to 0.5 Å/s for a mixture of Ar/N_2 = 2/1. The substrate temperature was varied between 150°C and 300°C. A bias voltage of -30 V and +10 V was applied.

The growth process has been studied *in-situ* by threee different scattering techniques. First, specular relectivity (XRR) for growth rate calculation. Second and third , large angle off-plane scattering in Bragg-Brentano geometry (XRD) and Grazing Incidence Grazing exit large angle in-plane Scattering (GIXS). The integrated peak intensities reveal information about the texture and crystallinity, and the exact positions of the Bragg peaks yield information about the lattice constants, off-plane and in-plane, repectively.

For all samples, the energy of the incident x-rays was monochromatized to 12.917 keV (λ = 0.961 Å).

	Power Ti (W)	Power Al (W)	Temperature (°C)	Gas ratio Ar/N	Bias (V)
G3_05	80	0	300	2/1	- 30
G2_01	80	5	300	2/1	- 30
G2_02	80	5	300	2/1	- 30
G2_03	80	5	150	4/1.2	- 30
G4_04	80	5	300	2/1	+ 10

Deposition parameters for the various samples investigated.

RESULTS

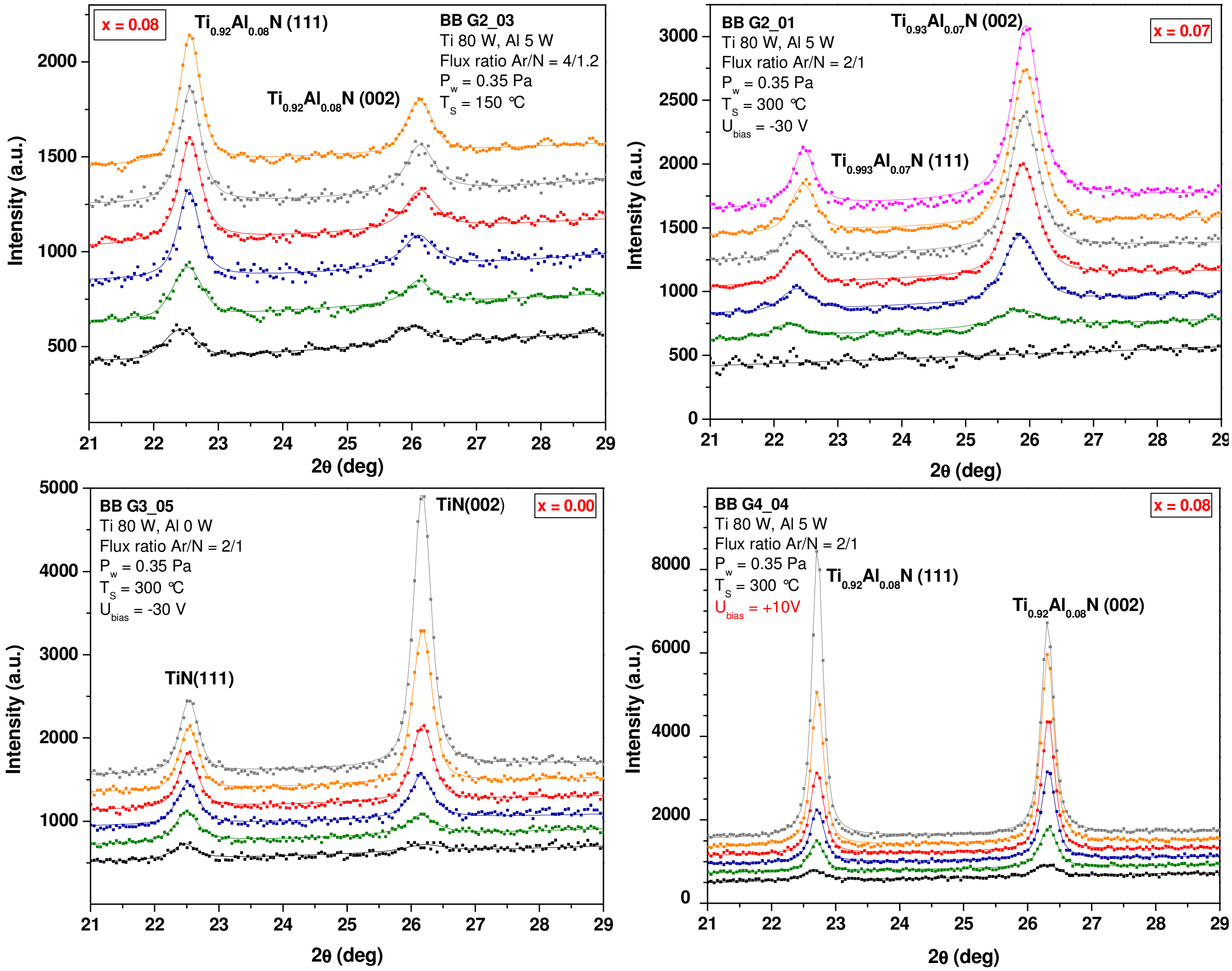

Fig 1: The dotted curves show the observed Bragg-Brentano intensities with increasing sample thickness (final thickness around 200–300 nm), the superposed straight lines show the fits obtained using a Pseudo-Voigt function for each single peak. The GIXS data show corresponding behaviour (*not shown*).

A deposition at 150°C and a growth rate of 1 Å/s results in a typical (111) preferred off-plane orientation for higher thicknesses (Fig. 1 *top left*). An increase of nitrogen partial pressure leads to stronger target poisening which in consequence leads to a lower deposition rate of 0.5 Å/s. This strongly effects the preferred off-plane orientation, which stays (002) even at higher thicknesses, practiacally regardless of substrate temperature (Fig. 1 *top right*). This behaviour cannot be attributed to the small amount of Al, since pure TiN shows exactly the same behaviour (Fig. 1 *bottom left*).

Because a reduced depositon rate also changes the ion/neutral-flux-ratio on the substrate, we suggest that the change in preferred orientation is not due to strain surface-energy competition but due to the increased ion bombardement during growth [3], and in fact suppressing the ion current by applying a positve bias reverses the texture into a competitive growth, where (111) orientation gets the upper hand at higher thicknesses (Fig. 1 *bottom right*).

[1] D. Gall, S. Kodambaka, M.A. Wall, I. Petrov, and J.E. Greene, J. Appl. Phys. **93** (2003) 9086
[2] W. Matz, N. Schell, W. Neumann, J. Bøttiger, and J. Chevallier, Rev. Sci. Instrum. **72** (2001) 3344
[3] M. Beckers, N. Schell, R.M.S. Martins, A. Mücklich, and W. Möller, *submitted* for publication