ROBL-CRG	**Experiment title:** *In-situ* **x-ray diffraction during sputter deposition of $Ti_{1-x}Al_xN$ – Part V: A new sputter chamber for MAX phase deposition**	**Experiment number:** **20_02_608**
Beamline: BM 20	**Date of experiment:** from: 31.08.2005 to: 03.09.2005	**Date of report:** 14.10.2005
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Report:

The heteroepitaxial growth of ***MAX phase*** Ti_2AlN ($M_{n+1}AX_n$ with M = Ti, A = Al, X = N and n = 1) ***on*** single crystal substrates ***MgO(111) and*** Al_2O_3***(0001)***, deposited by reactive magnetron co-sputtering from Ti and Al targets in an Ar/N_2 atmosphere at a temperature of >***850°C***, has been studied *in situ*. Using real-time specular x-ray reflectivity, ***layer-by-layer growth*** first of an approximately 10 nm thick epitaxial fcc $Ti_{0.63}Al_{0.37}N$ seed layer, then, after changing the deposition parameters, of the MAX phase itself was observed. Using off-plane Bragg-Brentano x-ray scattering, ***basal plane growth*** on both substrates can be deduced. ***Annealing up to 1200***°C gives a first indication of the ***phase stability of thin film MAX phase*** Ti_2AlN.

EXPERIMENTAL

In order to improve the ***base pressure down into the range of 10E-7 mbar*** (thereby minimizing any involuntary oxidation of the growing films) and in order to enlarge the accessible x-ray scattering range (in-plane and off-plane), a new magnetron sputter depositon chamber with ***larger Be-window openings*** was commissioned at the material research station of ROBL (Fig. 1).

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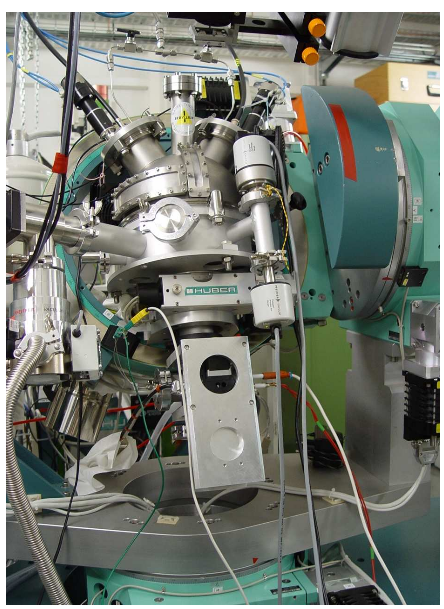
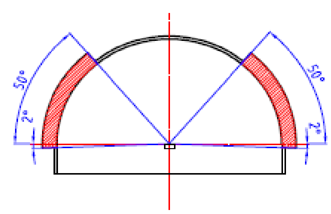
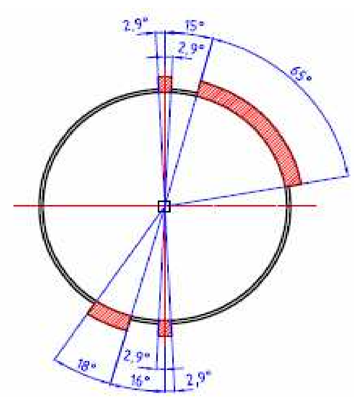


FIG. 1: Improved sputter depositon chamber with larger window openings made of Beryllium installed into the 6-circle ROBL diffractometer.



vertical scattering geometry



horizontal scattering geometry

A constant bias voltage of -30 V was applied for all depositions. The base pressure at the deposition temperature of ~850°C was ~2 x 10^{-6} Pa. For the fcc $Ti_{1-x}Al_xN$ seed layer an Ar/N_2 flux of 13.8/6.9 sccm was chosen leading to a working pressure of 0.35 Pa. The Ti and Al magnetron powers were 60 W and 20 W, respectively, leading to a composition of $Ti_{0.63}Al_{0.37}N$ as near as possible to the nominal corresponding MAX ratio. In order to achieve stable growth conditions for the Ti_2AlN MAX phase layer the deposition pressure was increased to 0.8 Pa at an Ar/N_2 flux of 39.7/2.4 sccm. The Ti and Al magnetron powers were 80 W and 26 W, respectively, leading to the Ti/Al ratio of 2/1 as required and calculated from preceding work.

The energy of the incident x-rays was monochromatized to 12.917 keV (λ = 0.961 Å).

Two different scattering geometries were employed: (1) low angle specular reflectivity either at a fixed incidence angle to determine the growth mode or scanned for the determination of the thickness; (2) large angle x-ray diffraction (XRD) in Bragg-Brentano geometry at stepwise increased temperatures up 1050°C in order to determine the off-plane lattice parameter and the phase stability, respectively.

RESULTS

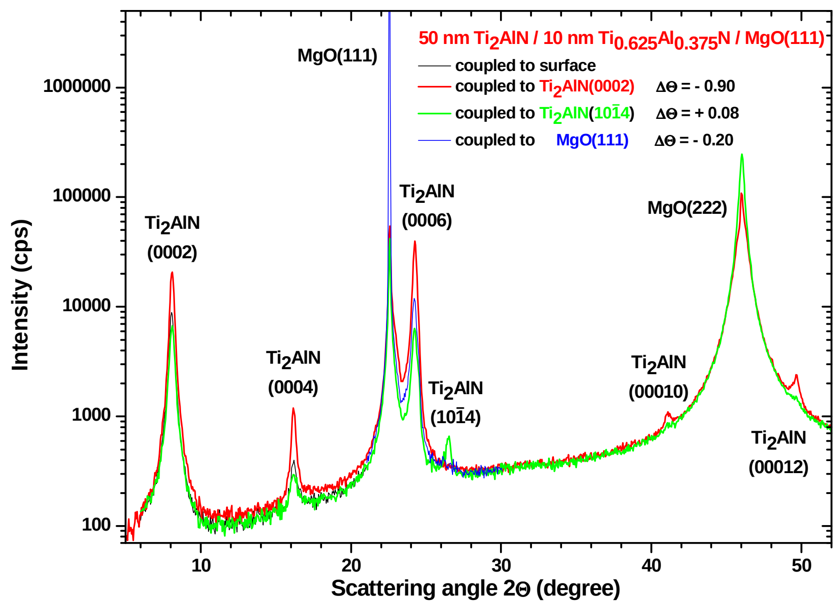


FIG. 2: *In-situ* XRD of the 10 nm thick fcc $Ti_{0.63}Al_{0.37}N$ seed layer and 50 nm thick MAX phase Ti_2AlN on the substrate MgO(111). At the higher deposition temperature of 850°C the multiplicity peaks (000x) are clearly seen over the now much larger angular range of more than 50° revealing ***basal plane growth*** in contrast to the *non*-basal plane growth at a deposition at 650°C (M. Beckers *et al.*, *Micro-structure and non-basal plane growth of epitaxial MAX Phase Ti2AlN thin films,* JAP (2005) *in press*, compare also Experimental Report 20_02_608 Part III).

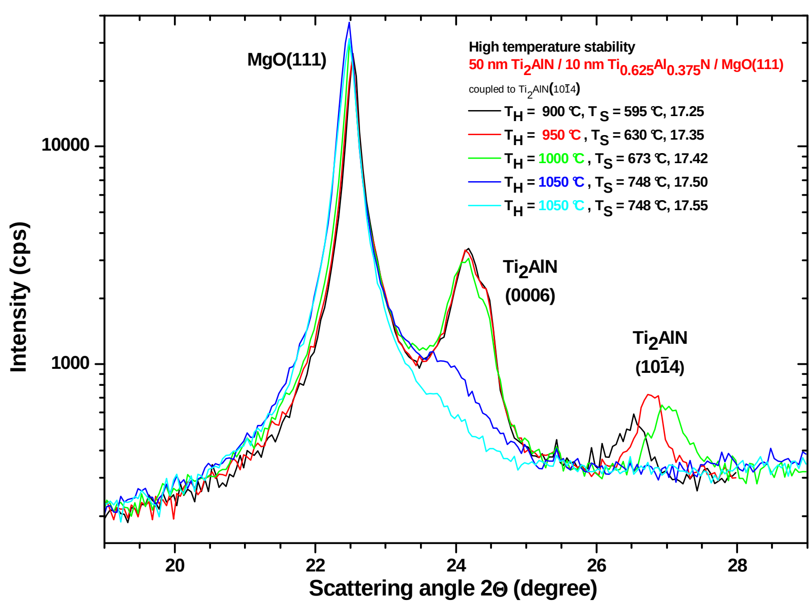


FIG. 3: ***Phase stability*** of a 60 nm thick MAX phase Ti_2AlN (on top of a 10 nm thick fcc $Ti_{0.63}Al_{0.37}N$ seed layer) on MgO(111) substrate : ***Above 1000°C the MAX phase disappears*** (probably by forming a spinell) indicating the tight phase diagram for the growth of perfect MAX phase thin films with their basal planes parallel to the substrate (as is necessary for future application).

On Al_2O_3 substrate the Ti_2AlN MAX phase is not prone to a spinell formation. However, it does not grow as well as on MgO (data not shown).