Experiment Report Form

The double page inside this form is to be filled in for each experiment at the Rossendorf Beamline (ROBL). This double-page report will be reduced to a one page, A4 format, to be published in the Bi-Annual Report of the beamline. The report may also be published on the Web-pages of the FZD. If necessary, you may ask for an appropriate delay between report submission and publication.

Should you wish to make more general comments on the experiment, enclose these on a separate sheet, and send both the Report and comments to the ROBL team.

Published papers

All users must give proper credit to ROBL staff members and the ESRF facilities used for achieving the results being published. Further, users are obliged to send to ROBL the complete reference and abstract of papers published in peer-reviewed media.

Deadlines for submission of Experimental Report

Reports shall be submitted not later than 6 month after the experiment.

Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report in English.
- include the reference number of the proposal / experiment to which the report refers.
- make sure that the text, tables and figures fit into the space available.
- if your work is published or is in press, you may prefer to paste in the abstract, and add full reference details. If the abstract is in a language other than English, please include an English translation.
- bear in mind that the double-page report will be reduced to 71% of its original size, A4 format. A type-face such as ”Times” or ”Arial” , 14 points, with a 1.5 line spacing between lines for the text produces a report which can be read easily.

Note that requests for further beam time must always be accompanied by a report on previous measurements.

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ROBL-CRG	**Experiment title:** Thermal stability and interface interactions in the TM-Al-N (TM = Ti, Cr) thin film nanocomposites	**Experiment number**: 20-02-684
Beamline: BM 20	**Date of experiment**: from: 21/10/09 to: 27/10/09	**Date of report**:
Shifts: 18	**Local contact(s)**: Carsten Baehtz	*Received at ROBL:*

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Report:

The development of the microstructure of $Ti_{1-x}Al_xN$ coatings during their treatment at elevated temperatures in vacuum was investigated by in-situ high temperature glancing angle X-ray diffraction (HT-GAXRD) experiments performed at ROBL BM 20. The samples were deposited using cathodic arc evaporation (CAE) at [Ti]/[Al] ratios between 60:40 and 33:67 and at bias voltages (U_B) ranging from -40 to -120 V. Preliminary laboratory experiments revealed that the initial microstructure, especially the phase composition in the as-deposited state, was modified via [Ti]/[Al] ratio and U_B [1]. At low U_B, fcc-(Ti,Al)N formed as a single phase up to x≤0.5. In contrast, the coatings deposited at high U_B contained fcc-(Ti,Al)N as major phase and AlN as minor phase. Our previous in-situ characterisation of the microstructure evolution of $Ti_{0.5}Al_{0.5}N$ coatings deposited at U_B=-40 V and U_B=-80 V during annealing at ROBL BM 20 (proposal numer; 20-02-675) revealed differences in the thermal stability of the coatings depending on the initial microstructure [2]. The main goal of the continuation of the previous study (20-02-675) was the investigation of the microstructure development during annealing for a wider range of chemical compositions (x=0.4, 0.5, 0.6, 0.67) and bias voltages (U_B=-40 V, U_B=-80 V and U_B=-120 V). Thus in-situ synchrotron HT-GAXRD experiments were done at 450℃, 650℃ and 850 ℃ as well as at 100℃ after each annealing step. From the analysis of the GAXRD patterns, the phase composition as well as the macroscopic lattice strain and the stress-free lattice parameter of the fcc-(Ti,Al)N phase were determined for all investigated temperatures. The results obtained from the GAXRD experiments are shown in figure 1 for the $Ti_{0.6}Al_{0.4}N$ coatings deposited at U_B = -40 V and

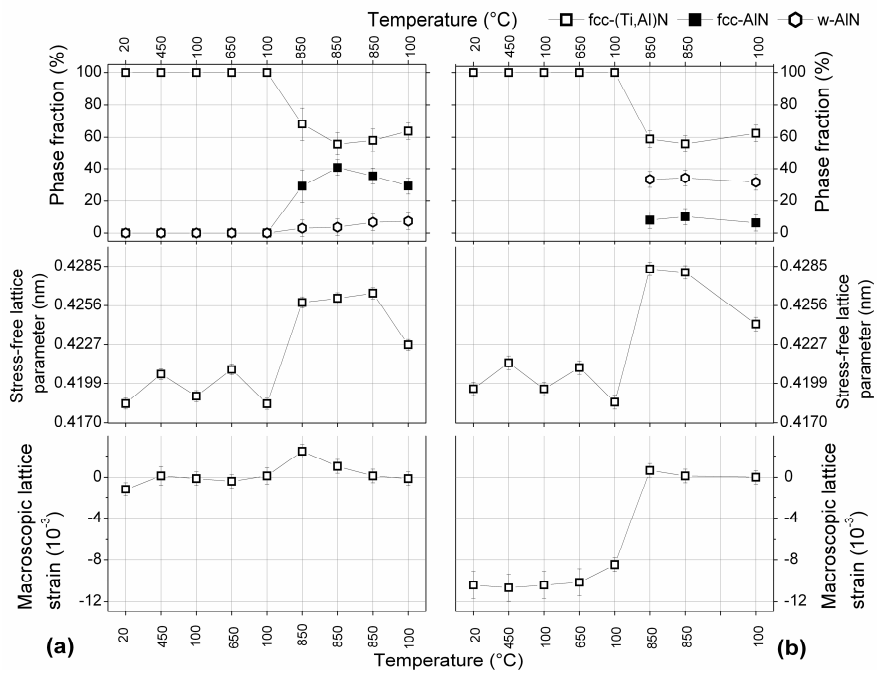

Figure 1: Evolution of the phase composition, stress-free lattice parameter of the fcc-(Ti,Al)N phase and macroscopic lattice strain of the fcc-(Ti,Al)N phase during annealing of $Ti_{0.6}Al_{0.4}N$ coatings deposited at U_B=-40 V (a) and U_B=-120 V (b).

-120 V. The analysis of the stress-free lattice parameter of the fcc-(Ti,Al)N phase (a_0) of the originally single-phase $Ti_{0.6}Al_{0.4}N$ coating (Fig. 1a) showed an increase of a_0 at 450℃ and 650℃ due to thermal expansion. After cooling from 650℃ to 100℃ a_0 returned to its starting value indicating that the coating remained single-phase up to 650℃. During annealing at 850℃, diffraction lines of w-AlN and fcc-AlN appeared in the GAXRD pattern. Rietveld analysis using the MAUD programme [3] yielded ~30 mol % fcc-AlN and ~10 mol % w-AlN. This onset of the decomposition was accompanied with an increase of both a_0 and macroscopic lattice strain in the fcc-(Ti,Al)N phase. Further annealing at 850℃ for ~100 min did not change the phase composition significantly. After cooling to 100℃, a_0 was smaller than 0.42418 nm, which is the lattice parameter of TiN. This indicated that some of Al was still incorporated in the fcc-(Ti,Al)N host structure; the coating consisted of ~60 mol% $Ti_{0.9}Al_{0.1}N$, ~30 mol% fcc-AlN and ~10 mol% w-AlN. In contrast to the originally single-phase coating (U_B=-40V), already ~ 30 mol% of w-AlN were formed and only ~10 mol% fcc-AlN could be observed in the dual-phase coating (U_B=-120 V) at 850℃. Further annealing at 850℃ did not change the phase composition. The analysis of a_0 after the 850℃ annealing step revealed that Al from fcc-(Ti,Al)N in the dual-phase coating $Ti_{0.6}Al_{0.4}N$ (U_B=-80V (not shown) and U_B= -120V (Fig. 1b)) segregated after 850℃ completely from the fcc-(Ti,Al)N host structure, because a_0 observed at 100℃ corresponds to Al-free fcc-TiN. The fraction of w-AlN and fcc-AlN in the $Ti_{0.6}Al_{0.4}N$ coating deposited at U_B=-80V was for both phases ~20 mol% and lay between the AlN fractions observed in the coatings deposited at U_B= -40V and U_B= -120V. The same tendency of the fraction of the decomposition products was observed in other investigated coatings.

[1] Ch. Wüstefeld, D. Rafaja, V. Klemm, C. Michotte, M. Kathrein, Surf. Coat. Technol. (2010), doi: 10.1016/j.surfcoat.2010.07.057.
[2] D. Rafaja, C. Wüstefeld, C. Bähtz, V. Klemm, M. Dopita, M. Motylenko, C. Michotte, M. Kathrein, Metal. Mater. Trans. A (2010), DOI: 10.1007/s11661-010-0204-8
[3] L. Lutterotti, D. Chateigner, S. Ferrari, J. Ricote, Thin Solid Films 450, (2004), 34-41.