The Rossendorf Beamline at ESRF

Forschungszentrum Dresden Rossendorf

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 of nanostructures in the Ti-Al-Si-N and Cr-Al-Si-N nanocomposites	**Experiment number**: 20-02/675
Beamline: BM 20	**Date of experiment**: from: 28/01/09 to: 31/01/09	**Date of report**: 08.07.2009
Shifts: 9	**Local contact(s)**: Carsten Baehtz	*Received at ROBL:*

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Report:

Changes in the microstructure of the $Ti_{0.50}Al_{0.50}N$ coatings due to their high-temperature treatment in vacuum were investigated by in-situ high-temperature measurements at ROBL beamline BM20. The samples were deposited by using cathodic arc evaporation (CAE) at two different bias voltages, U_B = -40 V and U_B = -80 V. Preliminary laboratory experiments revealed differences in the phase composition of the samples. $Ti_{0.50}Al_{0.50}N$ deposited at U_B = -40 V contained fcc-(Ti,Al)N as a single nanocrystalline phase. $Ti_{0.50}Al_{0.50}N$ deposited at U_B = -80 V contained fcc-(Ti,Al)N as the dominant phase and wurtzitic w-AlN as a minor phase. The crystallite size was about 7 nm and 3.5 nm for U_B = -40 V and U_B = -80 V. HRTEM experiments have shown that AlN was grown hetero-epitaxially on fcc-(Ti,Al)N. The main goal of the in-situ synchrotron experiments was to investigate the development of the phase composition in the $Ti_{0.50}Al_{0.50}N$ coatings and the changes in the stress-free lattice parameters and in the microstrain of fcc-(Ti,Al)N with increasing temperature up to 1140℃. Because of the expected spinodal decomposition, the coatings were cooled to approx. 110℃ after each high-temperature measurement and measured again. The results of the in-situ experiments are summarised in Fig. 1. The originally single-phase $Ti_{0.50}Al_{0.50}N$ coating remained single-phase up to approx. 660℃. In this temperature range, the stress-free lattice parameter increased slightly as driven by thermal expansion. After cooling the sample from 660℃ to 110℃, the stress-free lattice returned to the starting value. After heating the sample to 885℃, the metastable Al-rich fcc-(Al,Ti)N formed. Its amount increased during the annealing time (150 min) up to approx. 13 mol %. Concurrently, thermodynamically stable w-AlN

developed at 885℃. During the annealing at 885℃, the mole fraction of w-AlN reached nearly 5 %. The onset of the decomposition of fcc-(Ti,Al)N into fcc-(Al,Ti)N and w-AlN was accompanied with an increase of the stress-free lattice parameter of fcc-(Ti,Al)N as observed after cooling the sample and with an increase of the microstrain in the fcc-(Ti,Al)N phase. The increase of the stress-free lattice parameter confirmed a disappearance of Al from the host structure of fcc-(Ti,Al)N; the increase of the microstrain is an evidence of the formation of local strain fields at the interfaces between the decomposed fcc-(Ti,Al)N and the Al-rich phases. During annealing at 1140℃, the metastable Al-rich fcc-(Al,Ti)N dissolved almost completely and the coating consisted of nearly Al-free fcc- (Ti,Al)N (according to its stress-free lattice parameter) and w-AlN. During the last cooling from 1140℃ to 90℃, the sample decomposed almost completely into w-AlN (45 mol %) and Al-free fcc-TiN (50 mol %). Only traces of the Al-rich fcc-(Al,Ti)N phase (5mol %) were observed. In the dual-phase coating deposited at U_B = -80 V, the decomposition of fcc-(Ti,Al)N into the Ti-rich fcc-(Ti,Al)N and w-AlN was much faster than in the coating deposited at U_B = -40 V. Approx. 50 mol % w-AlN formed already at 890℃. The disappearance of Al from the host structure of fcc-(Ti,Al)N was confirmed by a large stress-free lattice parameter that was observed after cooling the sample from 893℃ to 115℃. The microstrain, which is an indicator of the intrinsic strain fields in the coatings, decreased continuously upon heating. Usually, large intrinsic strains are considered to increase the hardness of the thin film nanocomposites. In this particular case, both improved high-temperature stability and a higher high-temperature hardness are expected for the originally single-phase coatings than for the dual-phase coatings. Our in-situ high-temperature synchrotron measurements done on CAE (Ti,Al,Si)N coatings have shown that the addition of Si improves further the high-temperature stability of the microstructure of the coatings.

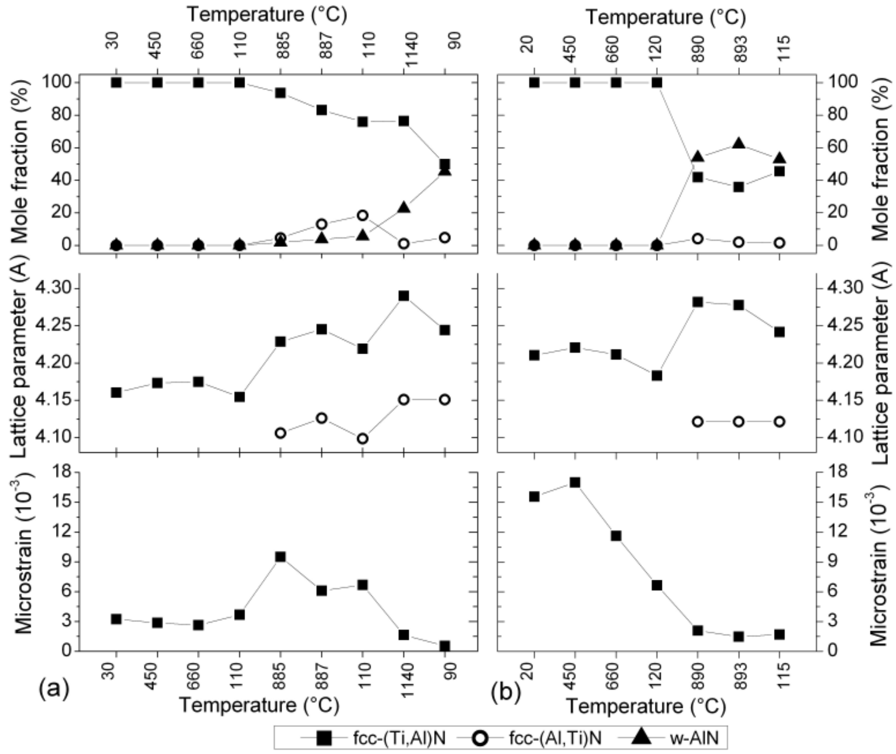

Figure 1: Phase evolution, stress-free lattice parameter and microstrain during annealing of the $Ti_{0.50}Al_{0.50}N$ coatings deposited at U_B=-40V (a) and U_B=-80V (b).