Forschungszentrum
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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:** Microstructure and interface interactions in Cr/ta-C multilayer thin films	**Experiment number**: 20-02-692
Beamline: BM 20	**Date of experiment**: from: 31/03/2010 to: 06/04/2010	**Date of report**: 28/01/2011
Shifts: 18	**Local contact(s)**: Dr. Carsten Baehtz	*Received at ROBL:*

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Report:

Microstructure development and the interdiffusion in Cr/ta-C multilayers during the heat treatment in vacuum were investigated using in-situ high temperature glancing angle X-ray diffraction (HT-GAXRD) and high temperature small angle X-ray (HT-SAXS) experiments performed at ROBL BM 20. The multilayers were deposited using a combined DC arc/laser arc technique, in which the Cr layers were deposited using the DC arc and the ta-C layers using the laser arc. The energy of the carbon ions was set to approximately 25 eV, 200 eV and 500 eV. Previous laboratory experiments showed that the carbon ion energy influences primarily the amount of sp^3 bounds in ta-C layers and the roughness of the Cr/ta-C interfaces. The main goal of the in-situ synchrotron experiments was to investigate the development of chromium carbides at the Cr/ta-C interfaces supported by the diffusion of carbon at higher temperatures. Within the Cr-C system, three stable carbides exist with narrow homogeneity ranges, which may work as diffusion barriers at higher temperatures. The samples were successively heated between 200°C and 600°C in steps of 100°C. The measurements were carried out after cooling the samples to 100°C to interrupt the carbon diffusion. The HT-GAXRD measurements showed the existence of the crystalline bcc Cr phase in all samples, whereas the carbon was amorphous. The sample with the carbon ion energy of 500 eV showed an additional peak corresponding to nanocrystalline chromium carbide. A more detailed in situ phase analysis was not possible because of the similar peak positions of all three Cr carbides. HT-XRR measurements showed that the carbon layer thickness decreased from 10.4 nm to 5.6 nm with increasing carbon ion energy, whereas the Cr layer thickness remained constant at approximately 10 nm. The heat treatment of the Cr/ta-C coatings at 200°C showed a recovery of the multilayer structure. The sample deposited using the highest carbon ion energy of 500 eV contained an additional layer with the thickness of about 2-3 nm, which might be related to the presence of a Cr carbide layer at the Cr/ta-C interface caused by a higher penetration of the C species into the Cr layer. Furthermore, this thin layer grows with increasing heat treatment to 5 nm at 200°C, which is related to the diffusion of carbon. The heat treatments at temperatures above 200°C resulted in a strong increase in the interface roughness for all Cr/ta-C multilayers. This confirmed a strong diffusion of carbon. The multilayer structure degraded for heat treatments at temperatures above 400°C. However, the degradation of the Cr/ta-C coating deposited at about 500 eV with strongly intermixed interfaces was less pronounced as compared to the other multilayers, which confirms the suggestion of the existence of one of the chromium carbides acting as diffusion barrier. The HT-SAXS measurements done on the multilayer deposited using 25 eV showed a roughness replication. The replication disappeared for the sample deposited using the highest carbon ion energy. Furthermore, the roughness replication decreased with increasing temperature of the heat treatment that corresponds to the increase in interface roughness.