

## 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.

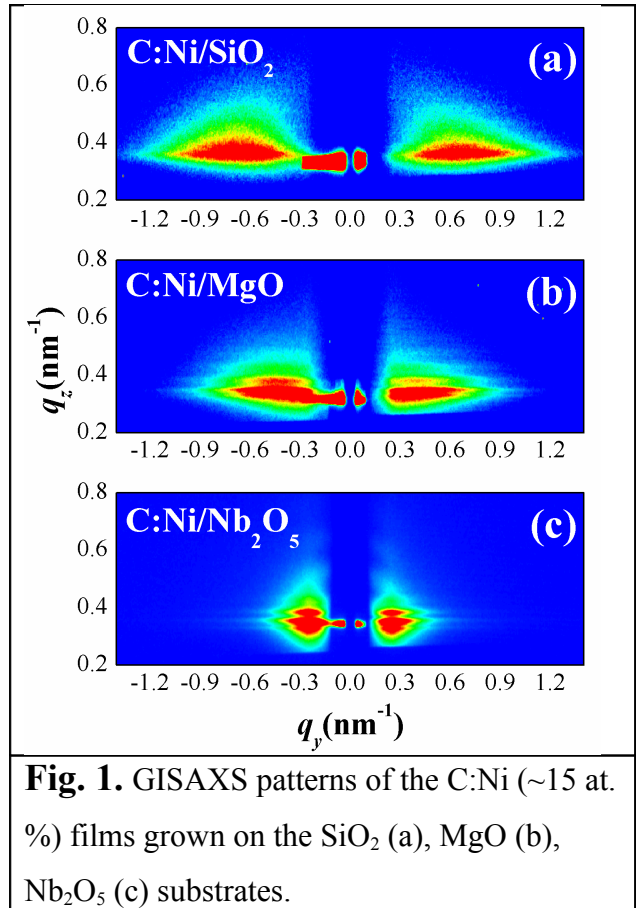
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|--|--|--|
| <br>ROBL-CRG  | <b>Experiment title:</b><br>Investigation of carbon encapsulated transition metal nanoparticles by the means of GISAXS | <b>Experiment number:</b><br>20-02-656 |
| <b>Beamline:</b><br>BM 20  | <b>Date of experiment:</b><br>from: 22/03/2008 to: 26/03/2008  | <b>Date of report:</b>                 |
| <b>Shifts:</b><br>12   | <b>Local contact(s):</b><br>Dr. Nicole Martha JEUTTER  | <i>Received at ROBL:</i>               |
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## Report:

The above experiments concern the investigation by the means of GISAXS of the metallic inclusion morphology in the carbon-transition metal (TM=V, Co, Ni, Cu) nanocomposite thin films grown by ion beam co-sputtering in the temperature range of RT-500° C. The obtained results allow establishing the following tendencies which are summarized in Table 1.

| System | Growth regime  |
|--------|--|
| C:V    | granular uncorrelated at low T → granular correlated at high T |
| C:Co   | granular uncorrelated at low T → columnar at high T            |
| C:Ni   | granular uncorrelated at low T → columnar at high T            |
| C:Cu   | granular correlated at low T → columnar at high T              |
|        |  |

It can be seen that for Co and Ni nanocomposites, there is a transition from a granular towards a columnar morphology. For C:Cu system, the growth at lower growth temperatures is granular but proceeds in a correlated manner, i.e. the renucleation occurs at relatively well determined distances and angles in relation to the substrate surface. At  $\sim 400^\circ\text{C}$ , there is a transition towards a columnar growth. For C:V nanocomposites, the growth proceeds in a un-correlated manner at low growth temperatures which undergoes a transition at higher temperatures towards correlated granular growth.



**Fig. 1.** GISAXS patterns of the C:Ni ( $\sim 15$  at. %) films grown on the  $\text{SiO}_2$  (a),  $\text{MgO}$  (b),  $\text{Nb}_2\text{O}_5$  (c) substrates.

The substrate type influence on the nanoparticle morphology was investigated for C:Ni( $\sim 15$  at.%) films grown at  $400^\circ\text{C}$ . At this temperature, the columnar growth takes place which is confirmed by the GISAXS results presented in Fig. 1. In addition, it is observed that the substrate type plays a crucial role on the nanocolumn diameter and interparticle distance. The results strongly suggest that the metallic islands formed at the initial stages of the film growth act as trap centers for the subsequently deposited metal adatoms. The results on the substrate type influence have been submitted to Journal of Physical Chemistry.

Despite the possibility to establish the tendencies of the nanoparticle growth, the measurement geometry needs to be re-optimized to cover the whole morphology range, as for the actual experiments very often the positions of the GISAXS spot maxima were either behind the beam stopper or out of the detector range. In addition, the intensity to noise ratio for C:V was rather poor which should be attributed to low density contrast of VC nanoparticles in relation to the carbon matrix.