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:** Investigation of chemical bonding structure and coordination of fullerene-like carbon-metal nanocomposite thin films by means of XANES and EXAFS	**Experiment number**: **20-01-662**
Beamline: BM 20	**Date of experiment**: from: 01/03/2007 to: 03/03/2007	**Date of report**: 03/04/2008
Shifts: 6	**Local contact(s)**: A. Scheinost	*Received at ROBL:*

Names and affiliations of applicants (* indicates experimentalists):

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Report:

Carbon-nickel (~30 at.%) nanocomposite thin films grown in the temperature range of RT-500°C have been investigated by the means of XANES and EXAFS at the ROBL beamline. The samples had been previously characterized by the means of transmission electron microscopy and x-ray diffraction. The latter shows that nickel is in the form of nanoparticles whose shape, size and phase depends on the growth conditions: Ni nanoparticles are nanorods with the diameter 3-10 nm in the temperature range of 500-300°C while lower temperatures prevent the columnar growth and particles exhibit granular shape. At 500-400°C, Ni is essentially in fcc metallic phase. When the temperature decreases, new hexagonal phase starts to form which becomes dominat at 300°C. For lower growth temperatures, no crystalline structure could be determined due to possible particle size effects or inherent amorphous structure.

The purpose of the EXAFS investigations was to get some information on the nearest neighbour coordination, local degree of atomic disorder and to identify possible

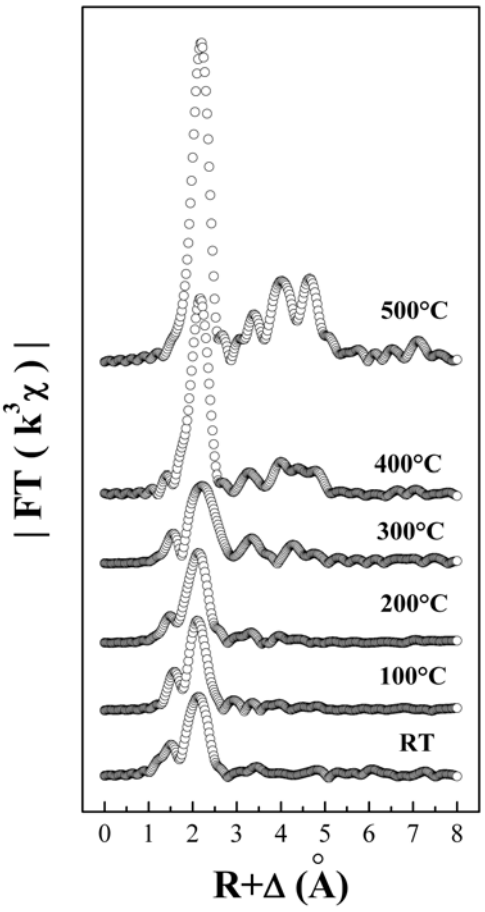

Fig. 1. The amplitude of the Fourrier transform of EXAFS oscillations of the C:Ni nanocomposites grown at RT-500°C.

size effects. The results are summarized in Fig. 1 which presents the amplitude of the Fourrier transform of the EXAFS oscillations of the C:Ni nanocomposites grown at different temperatures. These amplitude variations represent the distribution of the nearest neighbours in the coordination shells. One can see at 500-400°C a highly ordered Ni phase forms with the well defined first Ni-Ni and, more importantly, higher coordination shells. This structure corresponds to fcc Ni. At 300°C, one can identify additional nearest coordination shell which is attributed to Ni-C bonds. This allows identifying that the hexagonal phase observed by x-ray diffraction should be attributed to nickel carbide. At this temperature, still higher coordination shells could be identified which shows certain degree of crystallinity. Further decrease in temperature results in significant drop of the intensity of the coordination shells higher than the first Ni-C and Ni-Ni. This indicates about significant degree of disorder present within the nickel nanoparticles. No significant variation in the Ni-C intensity allows to demonstrate that indeed this effect cannot be attributed to the size effects but to the internal structure of Ni nanoparticles. These tendecies have been confirmed in the XANES spectra of the corresponding films.

In summary, EXAFS and XANES investigations have allowed to get deeper insights in the atomic structure of the Ni nanoparticles of C:Ni nanocomposite films. Thes results have been correlated with the magnetic properties. A manuscript has been submitted and now is in the review process.