## European Synchrotron Radiation Facility

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON



## **Experiment Report Form**

# The double page inside this form is to be filled in by all users or groups of users who have had access to beam time for measurements at the ESRF.

Once completed, the report should be submitted electronically to the User Office via the User Portal:

https://wwws.esrf.fr/misapps/SMISWebClient/protected/welcome.do

#### Reports supporting requests for additional beam time

Reports can be submitted independently of new proposals – it is necessary simply to indicate the number of the report(s) supporting a new proposal on the proposal form.

The Review Committees reserve the right to reject new proposals from groups who have not reported on the use of beam time allocated previously.

#### Reports on experiments relating to long term projects

Proposers awarded beam time for a long term project are required to submit an interim report at the end of each year, irrespective of the number of shifts of beam time they have used.

#### **Published papers**

All users must give proper credit to ESRF staff members and proper mention to ESRF facilities which were essential for the results described in any ensuing publication. Further, they are obliged to send to the Joint ESRF/ ILL library the complete reference and the abstract of all papers appearing in print, and resulting from the use of the ESRF.

Should you wish to make more general comments on the experiment, please note them on the User Evaluation Form, and send both the Report and the Evaluation Form to the User Office.

#### **Deadlines for submission of Experimental Reports**

- 1st March for experiments carried out up until June of the previous year;
- 1st September for experiments carried out up until January of the same year.

#### **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 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.

<b>ESRF</b>	Experiment title: Ion-assisted self-organization during the growth of carbon-transition metal nanocomposite thin films: a GISAXS study	Experiment number: 20-02-707
Beamline:	Date of experiment:	Date of report:
	from: 13/04/2011 to: 16/04/2011	31/01/2011
Shifts:	Local contact(s):	Received at ESRF:
	Dr. Carsten Baehtz	
Names and affiliations of applicants (* indicates experimentalists):		
Gintautas Abrasonis <sup>1,*</sup> ,Matthias Zschornak <sup>1,*</sup> , Sebastian Wintz <sup>1,*</sup> , Jose Luis Endrino <sup>2,*,†</sup>		
<sup>1</sup> Helmholtz-zentrum Dresden-Rossendorf e.V., P.O.Box 51 01 19, 01314 Dresden, Germany.		
<sup>2</sup> Instituto de Ciencia de Materiales de Madrid, Consejo Superior de Investigaciones Científicas, E-28049 Madrid, Spain.		
$\dagger^{\dagger}$ participated in the experiments, but is not listed in the application for the beamtime		

### **Report:**

Self-organized nanostructures in C:Ni nanocomposite thin films have been investigated by grazing-incidence small-angle x-ray scattering (GISAXS). New 2D Pilatus detector has been employed to detect the scattered GISAXS intensity. The C:Ni films were grown by ion beam assisted deposition (IBAD). During IBAD, the energy and momentum input into the growing C:Ni films is delivered by oblique incidence low energy (50-140 eV) Ar<sup>+</sup> ion beam. The energy and flux of the ion beam can be varied independently. During thin film growth, atomic displacements are caused by impacting energetic ions, resulting in phase separation in an advancing surface layer. Some samples were deposited on hot substrates (300°C and 500°C) to investigate the interplay of temperature induced thermal mobility and ion induced ballistic effects.

For the films with low Ni content (~15 at.%), the results demonstrate that low energy (~50 eV) ion assistance induces strong anisotropy in the film density distribution, i.e. in nickel rich and carbon rich regions (Fig. 1). The effect persists and is even more enhanced

for higher deposition temperatures. Transmission electron microscopy (TEM) studies show that such GISAXS structures correspond to Ni columns which are tilted in relation to the film

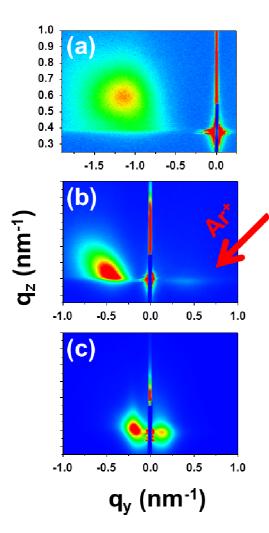


Fig. 1. GISAXS patterns of C:Ni(~15 at.%) nanocomposite films grown with ~50 eV  $Ar^+$  assistance at RT (a), 300°C (b) and 500°C (c).

surface. Morevoer, GISAXS allowed us establishing the orientation of the columns in relation to the ion beam which shows that the nickel nanocolumns tilt towards the incoming ions. For higher Ni atomic ratios the tendency is inversed.

Ion beam energy of ~130 eV results in sharp well-defined features in GISAXS patterns, some of them showing complex ripple-like intensity patterns (not shown). This is a manifestation of ion induced ordering processes during the film growth. It must be noted that these sharp features disapear if the sample is azimuthally rotated by 90°. This points out that the ion induced ordering is in the direction contained in the ion incidence plane. The origin of these features is currently under investigation and will be presented elsewhere.

From the fundamental point of view this study shows that phase separation processes at the nanoscale may be externally controlled assisting ion

beam and substrate temperature. Therefore, ion beam assisted deposition can be considered as a promissing tool to grow oriented and ordered composite nanostructures.