ESRF

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 using the **Electronic Report Submission Application:**

http://193.49.43.2:8080/smis/servlet/UserUtils?start

Reports supporting requests for additional beam time

Reports can now 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.

ESRF	**Experiment title:** Spontaneous ordering of Co nanoparticles in external magnetic fields.	**Experiment number**: SI1020
Beamline: ID10B	**Date of experiment**: from: June 23, 2004 to: June 29, 2004	**Date of report**: August 26, 2004
Shifts: 18	**Local contact(s)**: Sean O'Flaherty	*Received at ESRF:*

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Report:

Within the proposal" Spontaneous ordering of Co nanoparticles in external magnetic fields", the ordering of colloidal Co nanoparticles under various deposition/drying conditions was studied using GISAXS. The Co nanoparticles have been prepared in colloidal solution by the thermolysis of dicobaltoctocarbonyls (Co_2 $(CO)_8$) in an Ar ambient. A combination of oleic acid and oleyl amine has been used as a surfactant, which controls the size and prevents agglomeration. The magnetic field separation technique has been used to produce a colloidal suspension of particles of the size of ~10 nm with ≤ 5% size dispersion. The colloidal solution was diluted in toluene. Fabrication of well-defined 2D or 3D periodic nanoparticles arrays has important implications for development of nanoparticle-based devices. Ordered arrays of nanoparticles show specific properties different from those of bulk or isolated particles. Therefore, a detail study of ordered arrays of Co nanoparticles using GISAXS was done as planned in our proposal.

We analyzed the monolayers of Co nanoparticles prepared by spin coating method in our previous GISAXS studies. We have shown that using spin coating it is possible to produce a monolayer of particles on a large area standard substrate. As confirmed by GISAXS the short range ordering of particles of hexagonal type was obtained using this method [1].

Our HR-SEM and TEM studies have shown that the ordering of magnetic Co nanoparticles can be affected by the details of deposition conditions and by the application of an external magnetic field. Application of external magnetic field perpendicular to the substrate surface results in formation of larger areas of well-ordered monolayer of Co nanoparticles. The SEM studies showed formation of larger ordered 2D clusters with slightly different orientation to each other. Therefore, we used GISAXS to study the arrays of particles formed by simple drying in air, drying in perpendicular magnetic field (B≥0.8 T) and by spin coating. The measured GISAXS spectra point at the different degree of ordering arrays prepared by different procedures. The results are currently being analyzed.

superstructures - rods- of Co nanoparticles when magnetic field is applied. The size, shape and orientation of rods depend first of all on the orientation of applied magnetic field. According to our studies magnetic field of B≥0.2 T was high enough for rods formation. We studied the structure of rods prepared in perpendicular or parallel magnetic field within this proposal. In Fig. 1 the formation of rods in magnetic field parallel to the substrate surface is presented. We assume from the SEM studies that the rods are composed of close packed arrays of Co nanoparticles. However, from SEM images only ordering of surface layer can be analysed reliably. We utilized the beam-time allocated to measure the GISAXS spectra of rods oriented parallel or perpendicular to the substrate surface. For array of rods parallel to the substrate surface (Fig. 1) also GISAXS spectra for different orientation of rods with respect to the incoming beam were measured (Fig. 2).

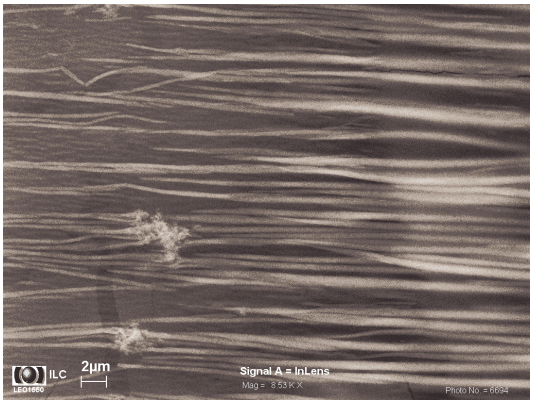

Fig. 1 HR-SEM image of 3D superstructures of Co nanoparticles in magnetis field 0.4 T parallel to the substrate surface.

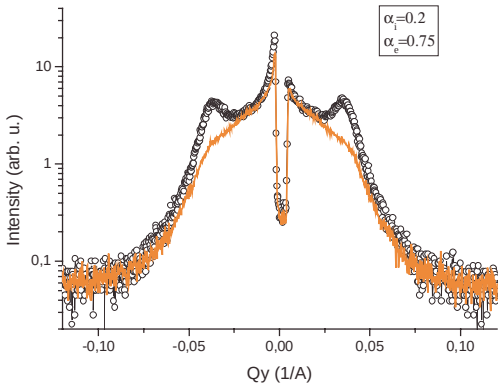

Fig. 2 GISAXS scans of 3D superstructures (Fig. 1) of rods parallel – and perpendicular – to the beam.

As the third type of experiment we performed the first measurments of the *in situ* GISAXS spectra of Co particles arrays formation during drying a drop in perpendicular magnetic field using a CCD camera.

All measured GISAXS spectra are analyzed and simulated at present. The preliminary analysis confirms that GISAXS is a very powerfull method for analyzing both- 2D and 3D arrays of nanoparticles. Especially 3D arrays representing magnetic mesostructures are very challenging as similar structures vere studied up to now only in in liquid state (ferrofluids). The GISAXS data will be combined with the analysis of magnetic properties to obtain a complex picture of these structures.

Reference

[1] Y. Chushkin, M. Ulmeanu, S. Luby, E. Majkova, I. Kostic, P. Klang, V. Holy, Z. Bochnicek, M. Giersig, M. Hilgendorff, T. H. Metzger, Structural study of self assembled Co nanoparticles, J. Appl. Phys. 94 (2003) 7743.