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: Spinodal decomposition of GaFeN investigated by anomalous x-ray scattering**	**Experiment number**: SI-1467
Beamline: ID01	**Date of experiment**: from: 25. 4. 2007 to: 1. 5. 2007	**Date of report**: 28. 8. 2007 *Received at ESRF:*
Shifts: 15	**Local contact(s)**: T. H. Metzger	

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Report:

The nature of ferromagnetic ordering of magnetic ions in epitaxial GaFeN and GeMn layers is not clear up to now. Unlike (Ga,Mn)As systems, for instance, where the ferromagnetic phase is observed only in heavily doped p-type layers, even undoped GaFeN layers exhibit ferromagnetic ordering [1,2]. Therefore, in addition to the hole-mediated spin ordering like in GaMnAs, another nature of the ferromagnetism must be present in these materials as well. Two possible explanations of this phenomenon exist in the literature, namely the presence of coherent Mn- (or Fe-) rich inclusions or incoherent ferromagnetic precipitates of other crystallographic phases. In [3] the possibility was discussed to tune the size of coherent inclusions in (Ga,Fe)N systems by changing the charge state of the magnetic ions by an intentional co-doping with shallow acceptors (Mg) or donors (Si).

The aim of this work was to study the structural parameters of inclusions in GaFeN and GeMn epitaxial layers by grazing-incidence diffraction and reciprocal space mapping. GaFeN layers have been grown by MOCVD method on sapphire substrates, the GeMn layers were prepared on Ge(001) substrates by MBE. For both sample types, a series of samples has been grown with various concentrations of magnetic ions and various growth parameters. X-ray diffuse scattering has been measured at a constant energy of 6.54 keV in grazing-incidence diffraction geometry (GID), in which the incidence angle α_i was kept constant. The diffracted intensity was measured by a linear detector perpendicular to the sample surface. Therefore, the detector signal represents the dependence of the scattered intensity on the exit angle α_f.

We found that the GaFeN samples contained many structural defects (mainly threading dislocations crossing the whole layer thickness and small-angle boundaries) giving rise to much stronger diffuse scattering than the Fe-rich inclusions themselves. Therefore, we focused our attention only to GeMn layers, where no structural defects (except for the inclusions) were present.

Figure 1 (upper row) shows the reciprocal space maps of three GeMn samples measured in the GID 220 diffraction. In the maps the axes q_r and q_a are parallel and perpendicular to the 220 diffraction vector respectively, the (q_rq_a) plane is parallel to the sample surface. The maps were measured with the incidence angle α_i = 0.3 deg just below the critical angle of total external reflection. From the measured data, a three-dimensional reciprocal-space distribution of the scattered intensity can be reconstructed; the maps shown in Fig. 1. depict only the scattered intensity integrated over the exit angle α_f. The samples #1, #2 and #3 differ in the nominal Mn content (3.4% in samples #1 and #3) and in the growth temperature (60°C for samples #1 and #3 and 120°C for sample #2).

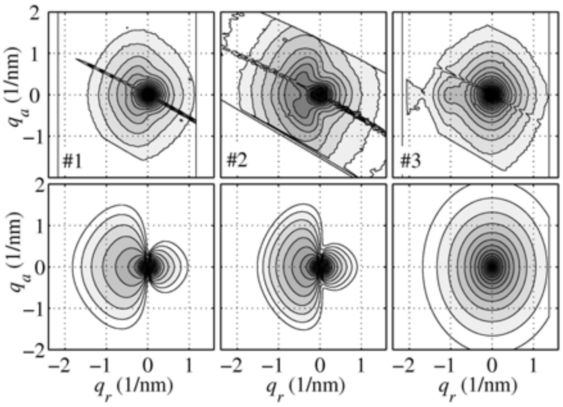

Fig. 1. Measured (upper row) and simulated (lower row) reciprocal-space maps of GeMn samples, GID diffraction 220

The central intensity maximum is the coherent crystal truncation rod of the substrate, for samples #1 and #2, the diffuse maximum is shifted radially towards the origin indication that the inclusion *expands* the surrounding lattice. A comparison of the measured and simulated intensity maps made it possible to estimate the inclusion radius to (1.2 ± 0.4) nm for samples #1 and #2, and (4.5 ± 0.2) nm for sample #3, as well as the mismatch between the inclusion lattice and the host lattice. For samples #1 and #2, we found the mismatch values (–3 ± 1)% and (–5 ± 1)%, respectively; the intensity map of sample #3 is almost symmetric, so that no deformation of the host lattice around the inclusions was detected.

From the measurements it follows that samples #1 and #2 contain coherent inclusions expanding the surrounding lattice. The inclusion size does not depend on the nominal Mn concentration in the layer; this concentration affects the mismatch, i.e., the enhancement of the concentration of the Mn atoms in the inclusion volumes. Sample #3 contains incoherent inclusions, containing the hexagonal Mn_5Ge_3 phase, most likely. The small side maximum in the intensity map of this sample (Fig 1., upper row) corresponds to the diffraction maximum 300 of this hexagonal phase.

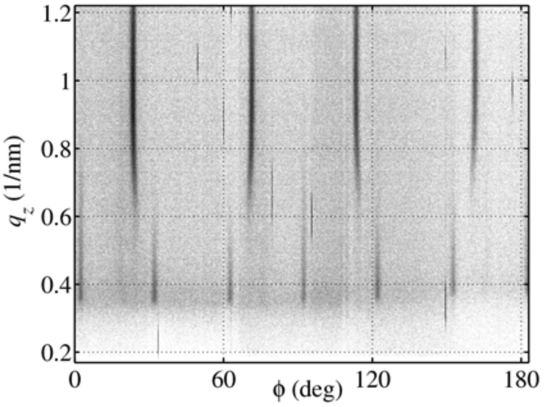

Fig. 2. Azimuthal map of sample #3, the 300 maxima lie at $q_z \approx 0.4$ Å^{-1}

Figure 2 shows a (φq_z) intensity map, where φ denotes the azimuthal position of the sample and q_z is the vertical component of the scattering vector. The 300 diffraction maxima repeat with the periodicity of $\Delta\varphi = 30$deg, which indicates that at least two different orientations of the hexagonal inclusion lattice exist in the layer.

Further investigation of the inclusion will focus on the dependence of the local mismatch of the inclusion lattice on the local Mn concentration. A publication of the results is expected in few months.

[1] T. Dietl, H. Ohno, F. Matsukura, J. Cibert, and D. Ferrand, Science **287**, 1019 (2000).
[2] T. Dietl, F. Matsukura, and H. Ohno, Phys. Rev. B **66**, 033203 (2002).
[3] T. Dietl, Nature Materials, in print (September 2006).