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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

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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.

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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:** Fe-rich nanocrystals in ferromagnetic GaFeN epitaxial layers	**Experiment number**: SI-1633
Beamline: ID-31	**Date of experiment**: from: 28.11.2007 to: 4.12.2007	**Date of report**: 27.2.2008 *Received at ESRF:*
Shifts: 18	**Local contact(s)**: Dr Alexander Dominic EVANS	

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Report:

GaFeN is a novel wide band gap semiconductor doped with a transition metal belonging to a new class of materials with magnetic properties whose origin and methods of control are not yet sufficiently well understood [1,2]. Most importantly, GaFeN exhibits Curie temperatures above room temperature. With SQUID magnetometry, apart from a paramagnetic behaviour, a ferromagnetic component of the magnetization curve is detected persisting up to 320K. The mechanism responsible for the ferromagnetic ordering of the spins of the Fe atoms is not yet fully established. It could be either mediated by carriers, it might also be a consequence of spinodal decomposition, or caused by foreign-phase inclusions. According to recent theoretical suggestions [3], the co-doping of GaFeN by donor (Si) or acceptor (Mg) impurities influences the position of the Fermi level in the energy gap and affects the charge state of the magnetic Fe^+ ions. Si doping increases the density of electrons which change the charge state of the iron ions from Fe^{3+} to Fe^{2+}. Whereas Fe^{3+} fits perfectly onto group III sites, the charges associated with the Fe^{2+} ions inhibit the creation of both coherent Fe-rich inclusions in the GaN lattice as well as the growth of foreign Fe-based phases.

The aim of the beamtime was to detect foreign phases in undoped, Si- and Mg-doped GaFeN layers, to determine the nature of these phases and to estimate the size of the precipitates. The measurements have been carried out at the ID-31 beamline using the energy of 15.5 keV, the diffracted radiation has been detected by a point detector equipped with a graphite secondary monochromator. We have measured a series of GaFeN layers with various Fe concentrations (achieved by various Cp_2Fe-flow rates in the MOVPE growth chamber) and with various concentrations of either Si or Mg dopants. For each sample, symmetric ω/2Θ scan and several ω scans across diffraction peaks were measured.

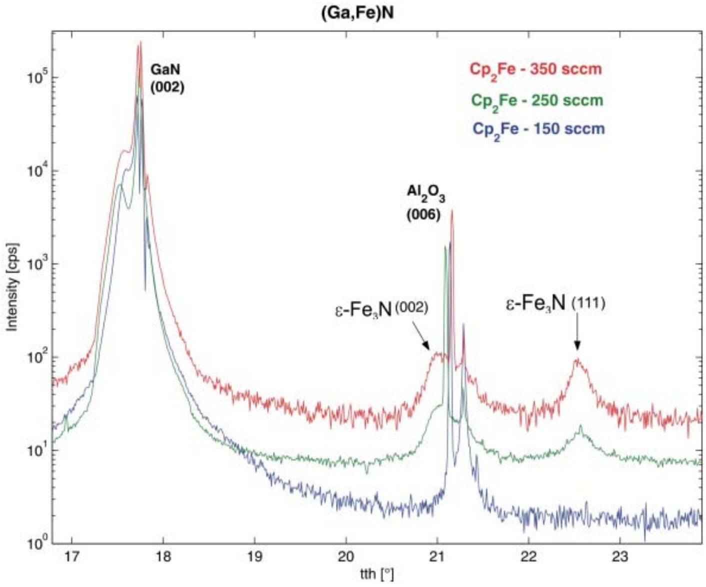

Fig. 1. Diffraction curves of undoped GaFeN samples grown with various Fe fluxes

Figure 1. shows an example of diffraction curves of undoped GaFeN layers with different Fe concentrations. For higher Fe contents, the solubility limit of Fe was exceeded, resulting in the formation of precipitates. In the diffraction curves, we have found two diffraction maxima of precipitates and have identified the these maxima as originating from ε-Fe_3N (hexagonal siderazot structure, PDF file #00-049-1663), in agreement with previous transmission electron microscopy studies [4]. From the FWHMs of these maxima estimated the mean diameter of the precipitates was estimated to be approximately 15 nm. Other, much narrower diffraction maxima in Fig. 1 were ascribed to the sapphire substrate and to diffractions from the GaN host lattice.

In Fig. 2 we present a comparison of the diffraction curves of undoped and Si-doped GaFeN layers.

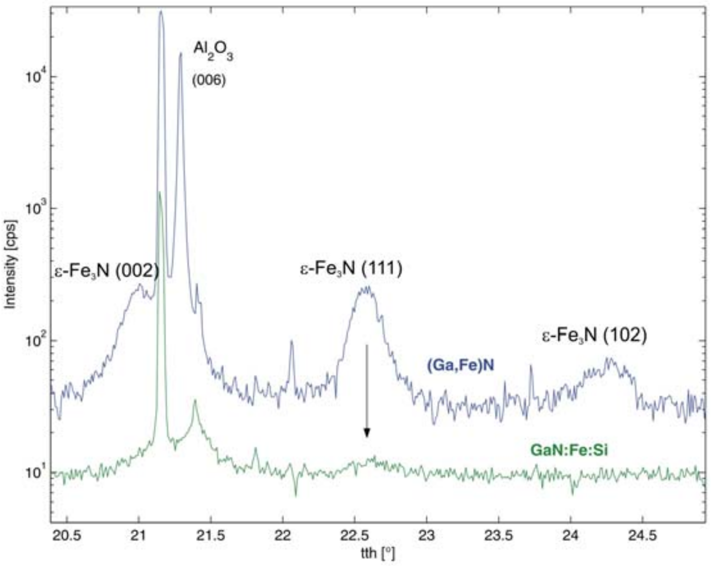

Fig. 2. Diffraction curves of undoped (blue) and Si-doped (green) GaFeN layers

From the results it clearly follows that codoping GaFeN with Si hinders indeed the growth of ε-Fe_3N precipitates.

So far only a preliminary conclusion can be drawn from diffraction curves of Mg-doped GaFeN layers, since the only GaFeN:Mg sample investigated up to now was not homogeneously Mg doped, but δ-doped, i.e. only a sequence of Mg doped layers separated by undoped regions were present. Again, ε-Fe_3N-related peaks disappear in Mg-doped samples. On the other hand, in these samples the presence of very small (few nm wide) inclusions of pure γ-Fe was detected; possibly caused by traces of γ-Fe on the sample surface as identified in TEM micrographs.

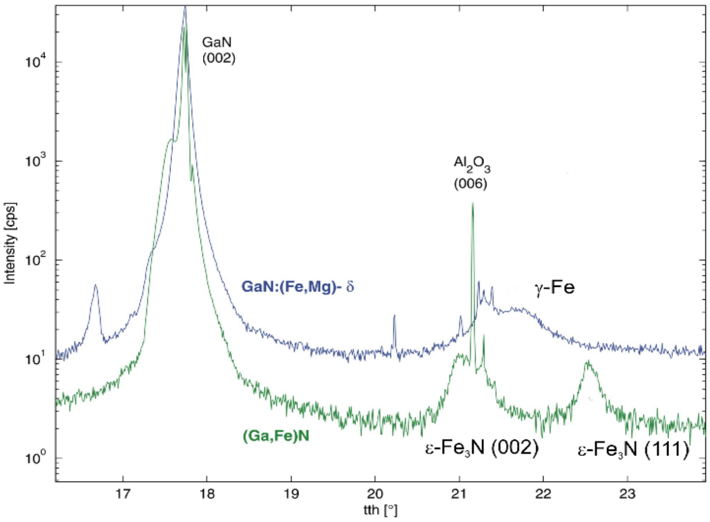

Fig. 3. Diffraction curves of undoped (green) und Mg δ- doped (blue) GaFeN layers

Summarizing, the diffraction curves measured during the SI-1633 experiment demonstrate for the first time that the codoping of GaFeN both by Si donor impurities hinders the creation of Fe-rich incoherent precipitates in the GaFeN matrix. This result is therefore a direct experimental proof of the hypothesis published in Ref. [3].

The role of acceptor impurities (Mg-ions) has to be studied further in homogeneously doped samples.

References

[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 **5**, 673 (2006).

[4] A. Bonanni, M. Kiecana, C. Simbrunner, Tian Li, M. Sawicki, M. Wegscheider, M. Quast, H. Przybylińska, A. Navarro-Quezada, R. Jakieła, A. Wolos, W. Jantsch, and T. Dietl, Phys. Rev. B **75**, 125210 (2007).