	Experiment title: The site of Fe in a new magnetic semiconductor: Fe:GaN	Experiment number: 08-01-751
Beamline: BM08	Date of experiment: <i>from:</i> 2006-11-25 <i>to:</i> 2006-11-28 <i>from:</i> 2007-06-14 <i>to:</i> 2007-06-17	Date of report: 2008-02-25
Shifts: 18	Local contact(s): Francesco D'Acapito	<i>Received at ESRF:</i>
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Report

X-ray Absorption Fine Structure (XAFS) experiment has been conducted at the Italian CRG beamline (GILDA / BM08) on a series of $Ga_{1-x}Fe_xN$ diluted magnetic semiconductors (DMS) in order to understand Fe local structure and incorporation in the host crystal as a function of deposition parameters. The main parameter studied here is Fe concentration (x) ranging around the solubility limit that in our growing conditions is $\approx 0.5\%$. Samples consists of $Ga_{1-x}Fe_xN$ films ($0.5 - 1 \mu\text{m}$) deposited by metalorganic chemical vapour deposition (MOCVD) on $1 \mu\text{m}$ GaN buffer layer and $\text{Al}_2\text{O}_3(0001)$ substrate.

The allocated 18 shifts for this experiment have been divided in two parts (November 2006 and June 2007) due to beamline technical problems. During these sub-experiments, spectra have been collected at Fe K-edge (7112 eV) in fluorescence mode using the monochromator equipped with Si[111] double crystals and Pd mirrors at high incidence (3.3 mrad) for harmonics rejection.

The first step of data collection has been focused on contaminations detection study using x-ray fluorescence to avoid systematic errors during the acquisition. In particular has been found an high amount of Fe contained in the kapton-tape glue used to fix samples on the sample holder, Gd (L_{III}-edge at 7243 eV) coming from fluorescence paper used in the sample alignment procedure and Co (K-edge at 7709 eV) coming from a not well identified source. In addition, we have also quantified the amount of Fe coming from the sapphire substrate on which the doped layers are deposited. After this preliminary investigation, experimental set-up has been optimized to avoid such contaminations in the fluorescence collected signal.

Investigated samples are reported in Tab. 1, where the 4** series consist of GaN:Fe single layer as described before and the 6** series consist of GaN:Fe multilayer (total thickness ≈ 200 nm) spaced by Mg δ -layers. EXAFS signals have been extracted with the ATHENA software (part of the well known IFEFFIT suite) after manual de-glitching of Bragg peaks (BPs) appearing in the fluorescence spectra due to reflections from the crystalline substrate. As visible in Fig. 1, this procedure has been possible only on the 4** series because collected spectra from the 6** series are completely distorted by BPs. In addition, in order to move the appearing BPs in a higher-energy region, spectra have been collected at different incidence angles (15 – 75 deg) but this procedure has been not useful in this case.

Sample	Fe (%)
465	0.4
472	0.6
470	0.7
471	0.9
466	1.0
664	1.0
665	0.2

Table 1: Investigated samples as function of Fe concentration (x) and deposition parameters.

The quantitative analysis has been carried out with the ARTEMIS software, with theoretical signals calculated using FEFF6L code. Fits presented in this report have been limited to nearest neighbours (NN) coordination distances, fitting in the R-space range [1.0 – 3.5] Å from the absorbing atom. Different theoretical models have

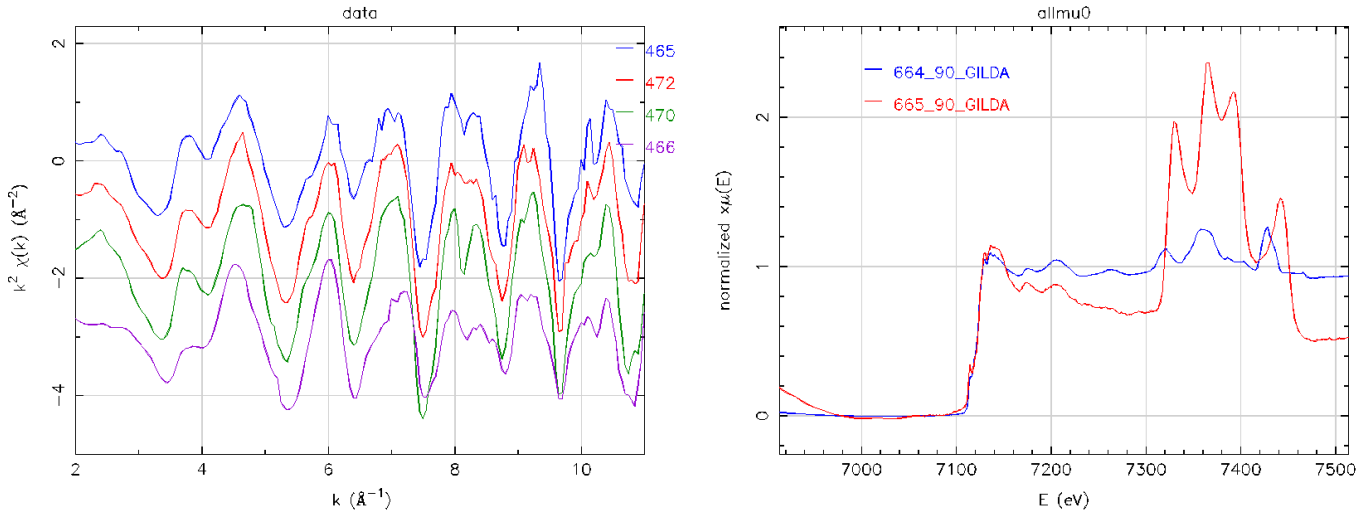


Figure 1: (left) EXAFS signal extracted from the collected spectra; (right) samples with Mg δ -doped layers presents distorted spectra impossible to analyze.

been tested before converging to the best-fit results illustrated in Fig. 2. To identify phases involved in the first-shell scattering, theoretical paths have been calculated from two models: 1) one substitutional Fe atom in (0, 0, 0) position in wurtzite GaN; 2) Fe_3N (Siderazot). The first model properly works for sample 465, showing a good Fe incorporation in the GaN host crystal. On the other hand, increasing Fe concentration, an additional peak in the Fourier-transformed spectra appears around 2.5 \AA as reported for sample 466 in Fig. 2. To obtain a good fit in this case, the first substitutional model needs to be completed with the second model; in particular the additional peak is fitted with an Fe-Fe contribution at $2.77(3) \text{ \AA}$. Contributions coming from Fe oxide or metallic are also tested without improvement of the fit.

In conclusion, this experiment has permitted to identify the solubility limit of Fe in GaN in our growth conditions with a fine description of the local environment around the dopant. From the preliminary first coordination shell analysis is possible to attest this limit at 0.5%: below this concentration samples presents Fe in Ga substitutional sites and above an additional phase appears: Fe_3N . This phase is well identified due to a characteristic distance of the Fe-Fe first neighbours (2.7 \AA) that rules out the presence of metallic or oxide Fe precipitations. In addition, this result is in line with previous transmission electron microscopy (TEM) characterizations. Unfortunately during this experiment has not been possible to study other deposition parameters like Mg co-doping (with δ -layers) due to distorted collected spectra; for this reason additional beam-time is desirable.

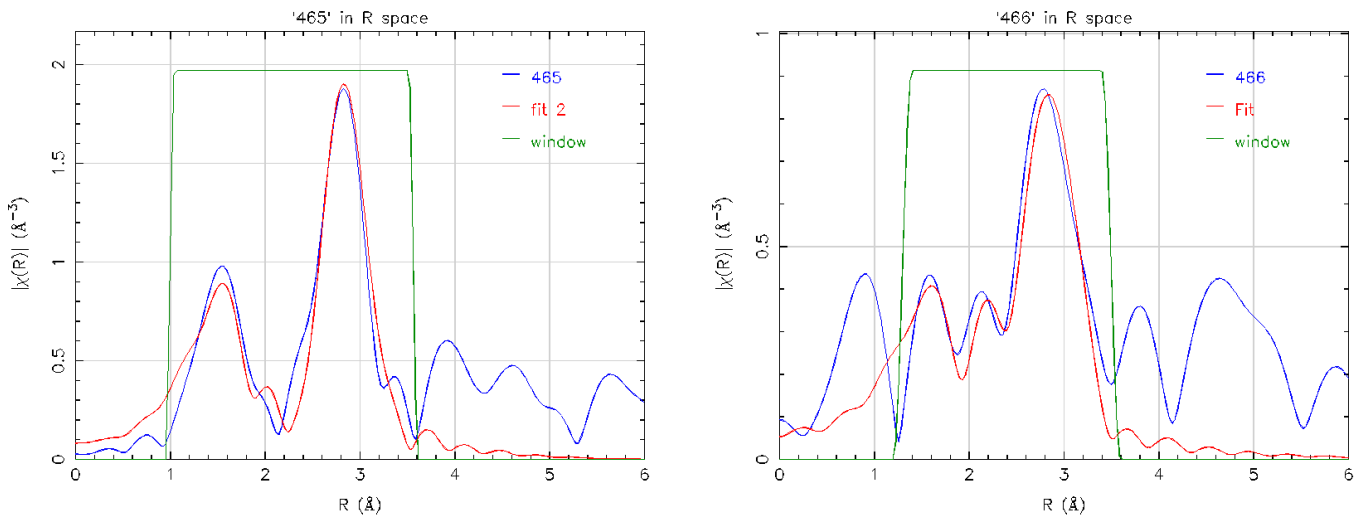


Figure 2: Fourier transform of the k^2 -weighted $\chi(k)$ (EXAFS) with an Hanning window in the k -range $[2 - 11] \text{ \AA}^{-1}$ and the relative I-coordination-shell best fits for sample 465 (left) and 466 (right).