ROBL-CRG	**Experiment title:** **In-situ x-ray diffraction investigation of growth of ferromagnetic clusters in (Ga,Mn)N epitaxial layers**	**Experiment number**: SI-1981
Beamline: BM 20	**Date of experiment**: from: 23/09/2009 to:03/00/09	**Date of report**: 24/02/2010
Shifts: 18	**Local contact(s)**: Nicole Jeutter	*Received at ROBL:*

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Report:

In contrast to (Ga,Fe)N, very little is known about (Ga,Mn)N. No attention has been paid so far to a possible growth of incoherent precipitates of other Mn-rich phases, which might affect the magnetic performance of prospective spintronic devices. We have hints that by increasing the concentration of the magnetic ions through the samples series, regions rich in Mn but coherent with the surrounding GaN matrix start to emerge.

Recently the fabrication (Ga,As)Mn layers with MnAs nanocrystals, for spintronic applications (e.g. spin-battery effect [1]), has been achieved through annealing of the dilute (Ga,Mn)As layers at temperatures ranging from 500 to 700°C[2].

For this purpose, two dilute samples of (Ga,TM)N (with TM=Mn, Fe) capped with Si_3N_4, were first measured in coplanar geometry at BM20 using a photon energy of 10 keV. Long radial scans from 10° to 45° in $\omega/2\theta$ were performed on each sample. Once it was established that the samples were dilute (*i.e.* did not present any secondary phases), they were annealed up to 900°C using the beryllium dome under nitrogen atmosphere and keeping a constant pressure of 200 mbar, in order to promote the aggregation of the TM ions.

The temperature was rapidly increased from 22 to 500°C, where long radial scans were taken after realigning with the GaN(002) peak. The samples were then heated up to 900°C, realigning after each 100°C step and taking long radial scans for each temperature step. However, no evidence of second phases formation during the entire annealing procedure was observed neither for the Fe nor the Mn doped GaN layers as can be seen in Fig. 1a).

The next step was to anneal samples containing secondary phases, in order to observe phase transformations and stability of the phases.

On Fig. 1b) we show the effect of annealing on (Ga,Fe)N layers containing different Fe-rich secondary phases.

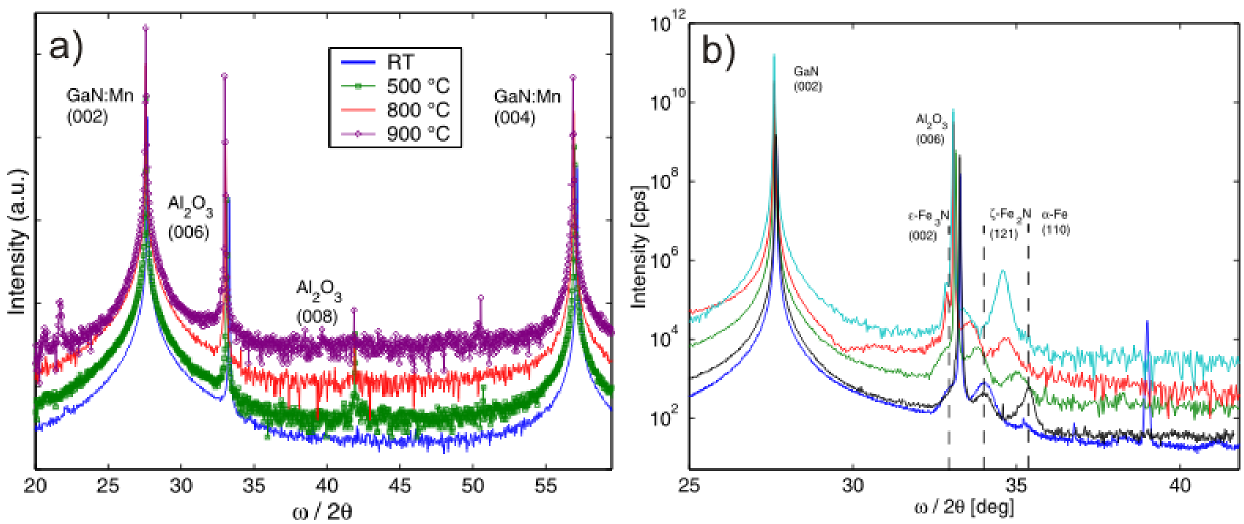

Fig 1. Annealing experiments on a) a (Ga,Mn)N dilute sample, and b) on a (Ga,Fe)N sample containing Fe-rich secondary phases.

As can be seen from fig.1 the most stable phase is ζ-Fe_2N, as it remains during the whole annealing process. The formation of α-Fe after annealing can be explained by the loss of nitrogen due to the heating, which promotes the formation of this Fe pure phase.

The measurements on the effect of annealing on the Fe_xN phases observed in (Ga,Fe)N performed during this beam time are under analysis and will soon be submitted for publication. Important parameters such as the change in lattice parameters (*d*-spacing) of the different phases, can be easily obtained from the obtained measurements.

References:

[1] P.Nam-Hai et al. *Nature* 07879 (2009).

[2] M. Tanaka et al. *Properties and Functionalities of MnAs/III–V Hybrid and Composite Structures,* Elsevier (2009).