Experiment title: *X-ray absorption and scanning X-ray micro-fluorescence study of highly Mn doped GaN*		Experiment number: HE1703
Beamline: ID21	**Date of experiment:** from: 01 April 2004 to: 06 April 2004	**Date of report:** 24 July 2004
Shifts: 15	**Local contact(s):** Remi TUCOULOU TACHOUERES	*Received at ESRF:*

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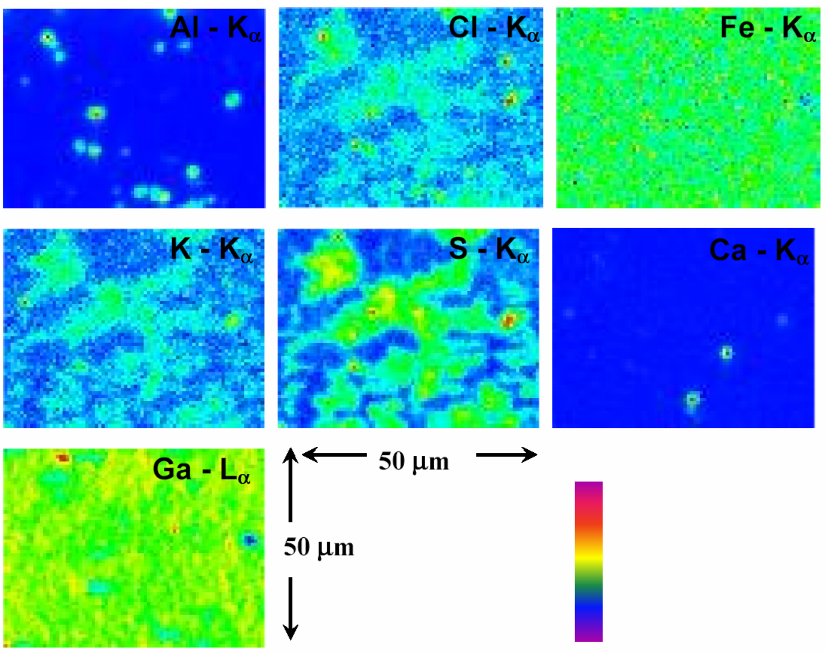
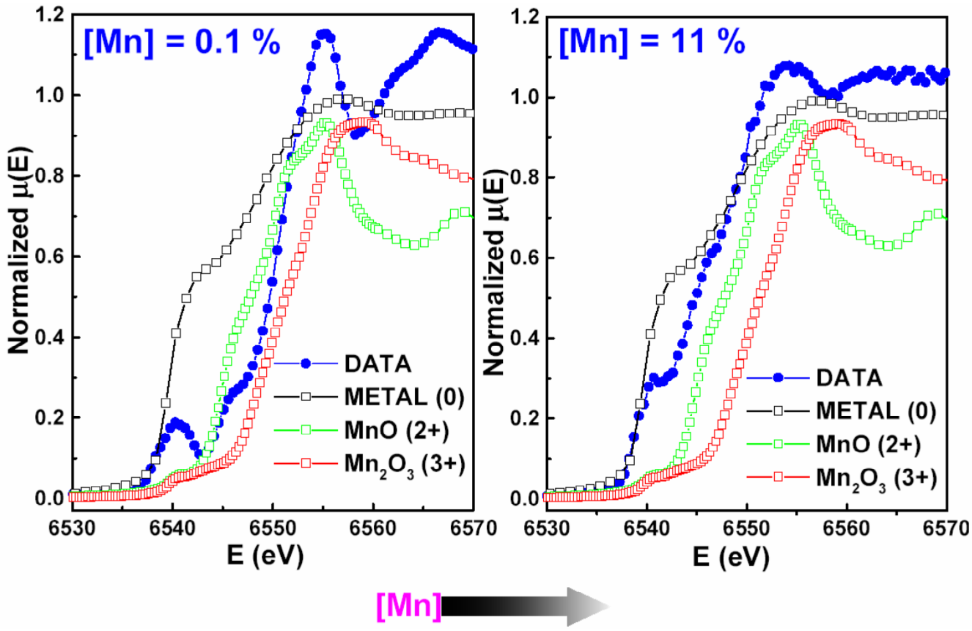
Report:

In this experiment, we studied highly Mn doped GaN samples deposited by MBE on sapphire (0001) substrates with a thickness of about 1 μm ([Mn] ranging from 8×10^{18} up to 5.4×10^{21} cm^{-3}). The access to K-absorption edges and fluorescence emission lines of the manganese and most common MBE background impurities, such as Si, Al, S, Ca, Fe, Cl, and K, has been possible as figure below shows for the highest doped sample. In close agreement with the previous results obtained at ID22, there is a co-localization of the principal residual elements and the Mn cluster distribution.



Generally, impurities can be incorporated during MBE GaN growth from the substrate material (sapphire in our case, Al_2O_3), effusion cell crucible (BN), memory effect of the growth chamber (Si doped GaN layers were formerly grown), and from the substrate cleaning procedure. Common background impurities in most of the MBE samples include C, Si, O and H. Carbon most likely incorporates on the N lattice site, acting like an acceptor in GaN. Silicon and oxygen, on the other hand, have been shown to be shallow donors in GaN. From the obtained elemental maps, we can conclude that the substrate cleaning process in our case played a dramatic role in the considered Mn-doped GaN samples. Usually, the surface grease is firstly eliminate by using C_7H_8O, and afterwards the residual oxides are removed by chemical annealing in a mixed solution: H_2SO_4:H_2O_2:H_2O (5:1:1). Similar procedures were likewise applied to get the best cleaning result. Summarizing, this result has given us an important feedback, and will be investigated further to improve the sample quality.

Thanks to the ID21 spectroscopic capabilities in the 2.5-7.5 keV range, the effective valence of Mn atoms and p-d hybridization was investigated by XANES at Mn K-edge:



While most experimental studies on the properties of the ferromagnetic semiconductor GaN:Mn have focused on isolated Mn substituting the Ga site (Mn-Ga), we investigate here whether alternate lattice sites are favored. As can be seen, there is an oxidation state shift as the Mn concentration increases in the GaN lattice from 2+ valence state towards 0+, suggesting the possible formation of a metallic secondary phase, e.g. Mn_4N. At the same time, the reduction and smoothing of the pre-edge peaks indicate a drop in the p-d hybridization, indicating the possible incorporation of Mn interstitials. Contrary to the expected dependence, as the transition metal incorporates into the lattice, the overlapping of its d-states and p orbitals of the host elements decreases suggesting a Mn site change. In summary, the ID21 submicron probes allowed us not only the possibility of residual elemental detection inside Mn-rich clusters, but also identification of doping-induced defects (secondary phases, residual impurities, interstitials). The effective valence of Mn atoms and p-d hybridization was studied in detail by XANES.