EUROPEAN ORGANIZATION FOR NUCLEAR RESEARCH

Proposal to the ISOLDE and Neutron Time-of-Flight Committee

Magnetic and structural properties of manganese doped (Al,Ga)N studied with Emission Mössbauer spectroscopy

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Abstract

Gallium nitride (GaN) and related compounds form a unique class of semiconductors with extraordinary qualities in terms of their crystal structure, optical properties, and electrical properties. These novel properties have made them useful in a wide range of applications in optoelectronic and high-frequency devices such as light emitting diodes, laser diodes and high power field effect transistors. When doped with a few percents of Mn and in the presence of free holes, GaN has been predicted to be a magnetic semiconductor with Curie temperature above room temperature. Mixed semiconductors of $Al_xGa_{1-x}N$ (AlGaN) composition, give rise to unexpected and critical magnetic and photonic functionalities when doped with magnetic ion species.

Here we propose an experiment on very thoroughly characterised AlGaN doped with Mn utilising extremely dilute ^{57}Mn ($T_{½}$=1.5 min), ^{57}Co ($T_{½}$ = 272 d) and ^{119}In ($T_{½}$=2.1 min) implantations, in order to perform ^{57}Fe and ^{119}Sn emission Mössbauer spectroscopy. The Mössbauer spectra give information on the electronic configuration of the implanted probe atoms and on magnetic properties, thus giving information on the physics of the materials at the most atomic-scale.

Requested shifts: 12 shifts, (split into ~3 runs over ~3 years)

1 INTRODUCTION/MOTIVATION

The search for dilute magnetic semiconductors has in the last decades been concentrated on (1) narrow band gap semiconductors (e.g. Mn doped GaAs), which are known to exhibit ferromagnetism, but have Curie temperatures below room temperature [Ohno96, Dietl10], and (2) wide band gap semiconductors (e.g. GaN and ZnO), where magnetism above room temperature in the presence of itinerant holes has been predicted by theory [Dietl00], but conflicting data on the magnetic properties have been reported [Dietl10].

Due to their unique photonic properties and the possibility of tuning the band gap over an exceptionally wide energy range, GaN and its alloys have advanced to the position of being considered as technologically outstanding semiconductors. Most commercial light emitting diodes (LEDs) are currently based on GaN, moreover, when homogeneously doped with transition metal ions and with a considerable amount of free holes, it has been predicted to be the ideal candidate for a ferromagnetic semiconductor with spintronic functionalities above room temperature [Dietl00]. Over the last years, it has been demonstrated that owing to specific features of magnetic impurities in semiconductors, the epitaxial growth of these systems can be reliably directed to result in: (i) a *dilute*, homogeneous alloy [Sawicki12], or (ii) a *condensed*, inhomogeneous system with the self-organised aggregation of magnetically robust nanocrystals (NCs) embedded in the host paramagnetic matrix, with specific signatures crucially depending on the crystallographic phase of the specific NCs [Bonanni08], or (iii) the formation of *complexes* involving one magnetic impurity and one or more electrically active dopant [Devillers12].

Specifically, over the last few years, by establishing a protocol of epitaxial growth and extensive characterization, it has been demonstrated that a strong hybridization and *p-d* interaction in GaN doped with transition metals give rise to remarkable magnetic, photonic, and electrical behaviours due to the small lattice parameters (compared to other technologically relevant semiconductors such as GaAs) [Navarro12, Suffczyński11, Bonanni11]. While magnetically doped GaN has been extensively studied, the investigations on AlGaN have been rather focused on this material as building blocks for LEDs, lasers and high electron mobility transistor (HEMT) structures.

The focus of the present proposal, however, are the electronic configurations of the dopant ions and their magnetic interactions with the host matrix since the wider band gap and even smaller lattice parameters of AlGaN compared to pure GaN are expected to extend the magnetic and photonic functionalities of the magnetically doped material.

To this end, the emission Mössbauer experiments proposed here will allow us to gain essential insight into the role played by Mn dopants in determining the properties of AlGaN:Mn systems.

2 SAMPLES AND METHODS

2.1 (Al,Ga)N:Mn samples

The samples consist of single-crystal wurtzite (wz) $Al_xGa_{1-x}N$ doped with Mn grown by metalorganic vapor phase epitaxy (MOVPE) on a 1 µm GaN buffer layer on c-plane sapphire. The doped layer is 600 nm thick. The samples are grown under H_2 atmosphere, with a pressure of 200 mbar and a temperature of 850°C. The samples have been subjected to extensive, detailed characterisation. The Mn concentrations considered in this work range from 0 to 5% as measured by secondary ion mass spectroscopy (SIMS). The absence of parasitic elements like hydrogen or oxygen is confirmed by SIMS, energy dispersive x-ray spectroscopy (EDX), Raman spectroscopy, and electron energy loss spectroscopy (EELS) measurements. The structure of the layers is characterised by high-resolution x-ray diffraction (HRXRD). In addition, high-resolution transmission electron microscopy (HRTEM) was performed on a JEOL 2011 Fast TEM microscope operating at 200 kV and capable of an ultimate point-to-point

resolution of 0.19 nm and allowing to image lattice fringes with a 0.14-nm resolution. The combination of the two techniques allows one to rule out the presence of precipitation in the layers.

2.2 Mössbauer spectroscopy

In this proposal, we want to make use of ^{57}Mn ($T_{½}$ = 1.5 min.) and ^{57}Co ($T_{½}$ = 271 d) for ^{57}Fe emission Mössbauer spectroscopy (eMS), and ^{119}In ($T_{½}$ = 2.1 min.) for ^{119}Sn emission Mössbauer spectroscopy, to study the structural and magnetic properties of Mn doped (Al,Ga)N. The use of Mn and In is ideal in the study of this system. After implantation, the ^{57}Mn adopts the lattice location of Mn (the III site) before decaying (β decay) to the Mössbauer state of isotope ^{57}Fe. The ^{57}Fe Mössbauer spectrum contains information on the magnetic hyperfine field that can be related to the spin polarization of the ^{57}Fe atom.

The main motivation for applying radioactive probe ^{57}Fe atoms for eMS in this project is that it allows to study the system with extremely dilute implantation of probe atoms (below ~10^{-4} at.%). For Mössbauer spectroscopy using stable ^{57}Fe or ^{119}Sn, concentrations above 10^{-1} at.% are required. Implantations to this level will render the material amorphous, and during high temperature post-annealing in order to remove crystal damage, precipitation cannot be avoided. Employing low-fluence radioactive implantations, high dose damage accumulation does not occur, and crystalline properties are maintained (see discussion below on specifically GaN and AlN). Furthermore, at Fe concentrations above 10^{-2} at.% spin-spin relaxations start to dominate, making interpretation on magnetic properties more complex.

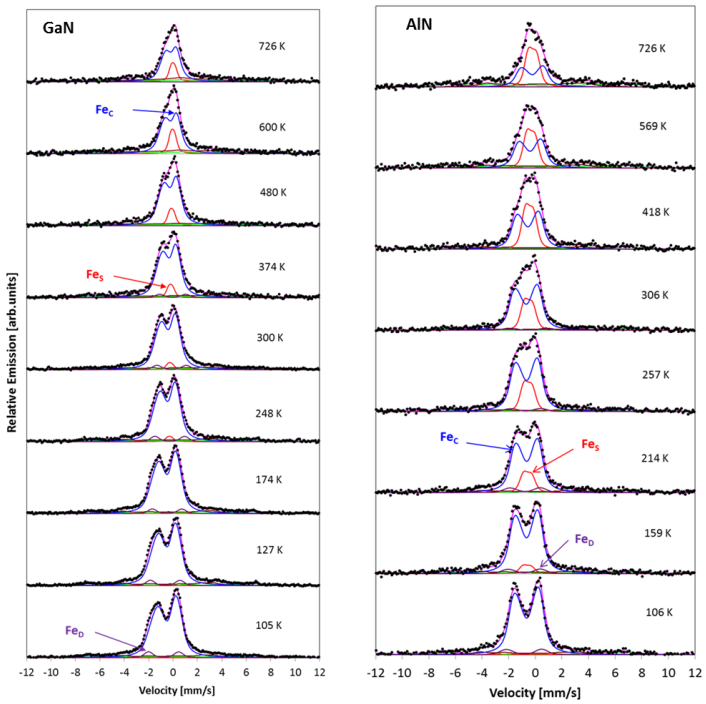

Fig. 1: ^{57}Fe Mössbauer spectra obtained after implantation of ^{57}Mn into GaN and AlN films on sapphire grown by hydride vapour phase epitaxy (HVPE), held at the temperatures indicated.

Utilising Mössbauer spectroscopy, information on the interactions of the probe atom with the nearest neighbour atoms can be obtained through the hyperfine interactions. This includes determination of the spin/valence state of Fe, site symmetry, and magnetic interactions. Slow paramagnetic relaxations and ferromagnetic ordering can be distinguished for Fe^{3+} [Gunnlaugsson10] and for measurements above the ordering temperature, fast spin-relaxations will be evidenced in the emission Mössbauer spectra [Gunnlaugsson12, Mølholt12].

We have already analysed ^{57}Fe Mössbauer data concerning the end-members (GaN and AlN) upon implantation of ^{57}Mn (cf. Fig. 1), and limited data on Fe doped GaN [H. Masenda, unpublished data].

After implantation, Fe atoms are found in environments dominated by point defects in their vicinity (Fe_C in Fig. 1). Angular dependent measurements (not shown) reveal that most probe ions are in crystalline environments and a small fraction in isolated amorphous zones (Fe_D) which is in accordance with results of emission channelling measurements [Wahl2001]. This demonstrates that the low implantation fluence used in these experiments preserves the crystallinity of the material.

As the sample temperature is increased during implantation and measurement, the point defects anneal, and the in-growth of defect free $Fe_{Al/Ga}$ (Fe_S in Fig. 1) is observed. The rate at which these processes take place is clearly different in the two materials. Moreover, the hyperfine parameters (position and splitting) are different in the two cases (cf. Table 1).

Additionally, the wings of the spectra show the presence of Fe^{3+} component exhibiting slow paramagnetic relaxations.

Table 1: Room temperature hyperfine parameters from fig. 1 obtained for Fe implanted in GaN (Black) and AlN (Red)

	Fe_S	Fe_C	Fe_D
δ (mm/s)	0.25(2) 0.51(2)	0.41(1) 0.68(2)	0.14(2) 0.56(3)
ΔE_Q (mm/s)	-0.30(3) -0.59(1)	1.21(2) 1.53(2)	2.35(3) 2.75(4)

3 EXPERIMENTAL PLAN AND BEAM REQUEST

At this point, it is envisaged that most of the data will be collected during two beam times, with the third beam time devoted to filling up the final gaps. If new unforeseen physics/properties are discovered, an addendum to this proposal will be proposed. The experimental plan outlined below will be revisited as data is obtained and analysed. The first steps in the investigation are relatively well defined, but subsequent steps will depend on the results obtained.

The first step of the experimental plan is to measure eMS spectra in a temperature series (90 K – 800 K). This yields information on annealing characteristics of the materials, the hyperfine parameters and site fractions as a function of temperature/composition. Data acquisition in each temperature series is estimated to take the order of 2-4 hours, depending on the complexity of the data and the beam intensity.

The goal is to obtain eMS data on about 5 different $Al_{1-x}Ga_xN$ compositions (~15 hours), 5 different AlGaN doped with ~3.5% Mn (~15 hours) and one AlGaN composition with varying Mn concentration (~15 hours). Initially, data will be collected on a few AlGaN compositions, subsequent compositions/temperature steps investigated will depend on the data already obtained.

At specific compositions/temperatures, angular dependent measurements with and without an external magnetic field will be conducted to verify lattice assignments and magnetic properties (~10 hours).

Depending on the results, specific compositions will be used for quenching experiments (implantation at elevated temperatures, measurements at lower temperatures), time delayed measurements [Gunnlaugsson09] and fluence dependence measurements (~10 hours).

^{57}Co implanted samples should be prepared on compositions where the interest is to study the magnetic behaviour in greater depth. Off-line measurements with long lived ^{57}Co allow for more varied sample environment than on-line measurements with the short lived isotopes (^{57}Mn and ^{119}In), and give possibilities of relevant post treatment.

We propose using ^{119}In ($T_{1/2}$ = 2.1 min) decaying to the Mössbauer state of ^{119}Sn, for these investigations also. Very limited data exists on ^{119}Sn in GaN for [Fanciulli95]. ^{119}In is expected to enter the III site (Ga or Al) [Weyer80], and is known to show significant dependence on ionicity within the group III-V semiconductors. ^{119}Sn has higher sensitivity to magnetic interactions than ^{57}Fe, and can be used to verify some of the findings obtained with ^{57}Mn implantations.

We would furthermore like to budget approximately one shift for calibration/contingency/opportunistic science/overhead. This will allow us (1) to follow up on new interesting and unexpected physics that will result from this investigation, (2) do comparative studies in related materials (such as Fe doped (Al, Ga)N and/or Mn doped GaAs), and (3) have room for non-proposed, but related studies. In many cases, members of the Mössbauer collaboration establish collaborations with researchers who are interested

in utilising the on-line Mössbauer method available at ISOLDE to investigate novel systems. These studies usually takes no more than 1-2 hours of beam time, which is not enough for a new proposal, but at the same time (often) results in good science, extended international collaboration, and consequent publications.

3.1 Complimentary studies

Where relevant, site assignments will be supported by theoretical calculations within the *ab initio* full potential LMTO method in the local density approximation (A. Svane).

The group of A. Bonanni has access to various options for post characterisation, including XMCD through collaborations with MaxLab in Lund and to ALBA in Barcelona. Additionally it should be stressed that samples implanted and measured at ISOLDE can be post-characterised with the range of techniques applied in the pre-implantation characterization (see above).

Complementary emission channelling experiments will be carried out on the exact same materials (cf. proposal presented to the INTC), making use of ^{56}Mn ($T_{½}$ = 2.6 h) and ^{59}Fe ($T_{½}$ = 44.5 d), from the parent ^{59}Mn ($T_{½}$ = 4.6 s), which are obtained from the same RILIS Mn beam as the ^{57}Mn ions requested in the current proposal. These lattice location experiments will greatly contribute to the interpretation of the here proposed emission Mossbauer experiments, in particular regarding site assignments.

3.2 Conclusions/Outlook

With the experimental plan as outlined above, we will have achieved the following:

1) Made detailed mapping of defect stability/evolution in (Al,Ga)N:Mn.
2) Made structural assignments to spectral components, possibly/likely assisted by theoretical calculations.
3) Identified the magnetic structure in (Al,Ga)N:Mn (fx. slow paramagnetism vs. ferromagnetism vs. fast spin relaxations)

The results of this study will provide

i) A body of firm data on the effects of the implantation process on the (Al,Ga)N:Mn system.
ii) An understanding of the physics of the differently doped systems that is essential in order to extend their range of applications.

Summary of requested shifts:

Isotope	Minimum Intensity/μC	Energy	Shifts	Target	Ion source
^{57}Mn (1.5 min)	(2-3)×10^{8}	≥ 50 keV	9	UC_x	Mn RILIS
^{119}In (2.1 min)	(2-3)x10^{8}	≥ 50 keV	2	UC_x	In RILIS[a]
^{57}Co (270 d)[ab]	9×10^{7}	≥ 50 keV	2	ZrO_2 or YtO_2	VADIS or Co RILIS
Total			13		

[*] If In RILIS is not available, surface ionization (W or Ta) can be a possibility.

^{57}Mn

^{57}Mn has been our work-horse for many years, decaying to the excellent Mössbauer state of ^{57}Fe (reasonable line-width and cross-section), and has been applied in many different scientific projects. In recent years, the Mössbauer collaboration has been able to use 5-6 shifts per beam-time without problems.

^{119}In

In 2010 surface ionized beam (W) of intensity ~(2-4)x10^7 enabled spectrum to be recorded within ~½ hour. In 2011, the ^{119}In intensity was too low to justify using the beam. According to U. Köster (private communication, 2011) and V. Fedosseev (private communication 2013) the ionization scheme for In is a simple two step process and should result in ~10 times more intensity. Moreover, the lasers can be optimized for the 2.4 minute isomer state of ^{119}In only.

^{57}Co

Although a long-lived isotope and despite commercially available sources of ^{57}Co, the number of facilities where ^{57}Co implantations are available have reduced in last decades. It is, however, of major interest, as it allows us to directly compare with the results obtained by ^{57}Mn. In 2010, a single sample was prepared with excellent dose, in 2011, few samples with minimum dose were prepared, and in 2012, the ^{57}Co dose was below the level of usefulness. If 2010 standard can be reached again, samples of ^{57}Co will become very useful in off-line studies.

4 ABOUT THE AUTHORS:

Austria: Institute for Semiconductor and Solid State Physics, Magnetic Spin Materials Group, Johannes Kepler University, Altenbergerstr. 69, Linz	A. Bonanni	Sample preperation (MOVPE) and structural and magnetic characterization methods including high-resolution x-ray diffraction (HRXRD), high-resolution transmission electron microscopy (HRTEM), electron energy loss spectroscopy (EELS), secondary-ion mass spectroscopy (SIMS), SQUID magnetometry, synchrotron x-ray absorption fine structure (EXAFS) and x-ray absorption near-edge structure (XANES).
Denmark: Department of Physics and Astronomy, Aarhus University, Ny Munkegade 120, DK-8000 Aarhus	H. P. Gunnlaugsson & A. Svane	Well equipped Mössbauer laboratory. Emission Mössbauer spectroscopy at *T*>20 K for long lived isotopes. Theoretical calculations of hyperfine parameters. Collaboration with the semiconductor group (A. N. Larsen) with optical characterization thechniques.
Italy: Laboratorio MDM, IMM-CNR, Via Olivetti 2, I-20864 Agrate Brianza (MB)	R. Mantovan	Mössbauer Lab. Growth of samples (ALD) and various characterization techniques.
South Africa: School of Physics, University of the Witwatersrand; School of Physics, University of KwaZulu-Natal, Durban 4001	H. Masenda, K. Bharuth-Ram, D. Naidoo, M. Ncube, M. Moodley	Mössbauer Labs., and experts in the analysis of Mössbauer data. Specialists in Nuclear Methods in Solid State Physics. Collaboration with Magnetization group at iThemba LABS, and Raman Spectroscopy group.
Iceland: Science Institute,	H. P. Gíslason, T.	Sputtering growth of various samples, with

University of Iceland, Dunhaga 3, IS-107 Reykjavík	E. Mølholt, S. Ólafsson	various types of characterization techniques. EPR expertize and analysis of Mössbauer data.
Switzerland: PH Department, ISOLDE/CERN, 1211 Geneva 23	K. Johnston, the ISOLDE collaboration	Radioactive Laboratory, equipment for annealing and on/off-line Mössbauer measurements.
Belgium, Instituut voor Kern- en Stralings fysika,KU Leuven	G. Langouche, L. M. C. Pereira	Mössbauer Lab., Specialist in Mössbauer spectroscopy (GL), ion implantation (stable), ion beam analysis, SQUID magnetometry, various other characterization techniques

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Appendix

DESCRIPTION OF THE PROPOSED EXPERIMENT

The experimental setup comprises: *(name the fixed-ISOLDE installations, as well as flexible elements of the experiment)*

Please note that in January 2014 a new collection chamber should be delivered to ISOLDE to replace the existing GLM chamber. The safety file will be updated in due course once the new chamber arrives. The existing chamber will remain as a "spare" and will be adapted as a removable system allowing collections to be performed on the HRS separator.

Part of the Choose an item.	Availability	Design and manufacturing
Mössbauer setup	☒ Existing	☒ To be used without any modification
SSP-GLM chamber	☒ Existing	☒ To be used without any modification ☐ To be modified
	☐ New	☐ Standard equipment supplied by a manufacturer ☐ CERN/collaboration responsible for the design and/or manufacturing

HAZARDS GENERATED BY THE EXPERIMENT

(if using fixed installation) Hazards named in the document relevant for the fixed [COLLAPS, CRIS, ISOLTRAP, MINIBALL + only CD, MINIBALL + T-REX, NICOLE, SSP-GLM chamber, SSP-GHM chamber, or WITCH] installation.

Additional hazards:

Hazards			
	Mössbauer setup (online experiments)	*SSP-GLM chamber*	*[Part 3 of the experiment/equipment]*
Thermodynamic and fluidic			
Pressure	Low pressure only	Low pressure only	
Vacuum	Yes	Yes	
Temperature	< 100°C (outside setup)	Room temperature	
Heat transfer	No	No	
Thermal properties of materials	Metal	N/A	
Cryogenic fluid	N_2, 1[Bar], 3 l/h	N/A	
Electrical and electromagnetic			
Electricity	<20 V, < 20 A	None	
Static electricity	None	None	
Magnetic field	< 0.1 T (outside setup)	None	

Batteries	☐		
Capacitors	☐		
Ionizing radiation			
Target material	Diverse	Diverse	
Beam particle type (e, p, ions, etc)	Ions	Ions	
Beam intensity	$<10^9$ s^{-1}	$<10^9$ s^{-1}	
Beam energy	>50 keV	>50 keV	
Cooling liquids	[liquid]		
Gases	[gas]		
Calibration sources:	☐		
• Open source	☐		
• Sealed source	☐ [ISO standard]		
• Isotope			
• Activity			
Use of activated material:			
• Description	☐		
• Dose rate on contact and in 10 cm distance	57Mn (estimated) @ 10cm 150 μSV/hr Similar for 119In Concrete shielding employed around implantation chamber	0.04 μSv/hr @ 10cm for 0.4MBq	
• Isotope	57Mn, 119In	57Co	
• Activity	~50 mCi (inside the setup) (185 MBq)	4-10 µCi (0.15-0.4 MBq)	
Non-ionizing radiation			
Laser			
UV light	No	No	
Microwaves (300MHz-30 GHz)	No	No	
Radiofrequency (1-300MHz)	No	No	
Chemical			
Toxic	No	No	
Harmful	No	No	
CMR (carcinogens, mutagens and substances toxic to reproduction)	No	No	
Corrosive	No	No	
Irritant	No	No	
Flammable	No	No	
Oxidizing	No	No	
Explosiveness	No	No	
Asphyxiant	No	No	
Dangerous for the environment	No	No	
Mechanical			
Physical impact or mechanical energy (moving parts)	No	No	
Mechanical properties (Sharp, rough, slippery)	No	No	
Vibration	No	No	
Vehicles and Means of Transport	No	No	
Noise			
Frequency	No	No	

Intensity	No	No	
Physical			
Confined spaces	No	No	
High workplaces	No	No	
Access to high workplaces	No	No	
Obstructions in passageways	Just outside GLM	No	
Manual handling	?	?	
Poor ergonomics	?	?	

Hazard identification

3.2 Average electrical power requirements (excluding fixed ISOLDE-installation mentioned above): *(make a rough estimate of the total power consumption of the additional equipment used in the experiment)*