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TO THE ISOLDE AND NEUTRON TIME-OF-FLIGHT EXPERIMENTS COMMITTEE (INTC)

STATUS REPORT AND ADDENDUM TO EXPERIMENT IS453

EMISSION CHANNELING LATTICE LOCATION EXPERIMENTS WITH SHORT-LIVED ISOTOPES

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SUMMARY

We report on the progress of experiment IS453. Since its commissioning in 2007 the new emission channeling setup of the EC-SLI collaboration has been used during three on-line beam times with radioactive Mn isotopes and recently also for ^{27}Mg. Since Septemper 2009 the setup is installed at the GHM beamline of GPS, which provides much more favourable conditions with respect to the required collimation of the ISOLDE beam than the LA1 or LA2 beam lines used previously. Successful experiments were performed determining the lattice locations of Mn and Co in ZnO and GaN, Mn in Ge and GaAs, and first exploratory results on Mg in GaN and AlN have been obtained as well.
We propose to continue the β^- emission channeling lattice location experiments using the transition metal probes ^{56}Mn (2.5 h), ^{59}Fe (45 d), ^{61}Co (1.6 h) and ^{65}Ni (2.5 h) implanted in several types of semiconductors, in particular ZnO, GaN, Si, Ge and GaAs. In addition, we would like to intensify the lattice location studies of the acceptor dopant ^{27}Mg (9.5 min) in the nitride semiconductors GaN, AlN, and InN. It is also planned to investigate the lattice location of the Li acceptor in ZnO by means of α emission channeling from the probe ^{8}Li (838 ms).

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1) Introduction

The principle of emission channeling [15] is to dope single crystals with radioactive probe atoms that decay by the emission of charged particles such as α, β^- or β^+ particles or conversion electrons, which, on their way out of the crystal, experience channeling or blocking effects along low-index crystal directions. The resulting anisotropic particle emission yield from the crystal depends in a characteristic way on the lattice sites occupied by the emitter atoms and is recorded with the aid of position sensitive detectors. In comparison to conventional lattice location techniques by means of ion beam channeling, e.g. Rutherford Backscattering/Channeling (RBS/C), the main benefit of emission channeling is a roughly four orders of magnitude higher efficiency. An additional and unique advantage is the fact that provided a suitable radioactive isotope is available there is no decrease of sensitivity regarding the detection of elements lighter than the host lattices, contrarily to RBS/C. These facts allow one to perform detailed lattice location studies with very good statistical accuracy at low fluences of implanted probe atoms, usually on the same sample as a function of post-implantation annealing temperature, which is not feasible by other methods.

In Sept. 2006 the ISOLDE Experiment IS453 "Emission channeling lattice location experiments using short-lived isotopes" (EC-SLI) was approved by the INTC and granted 20 of 36 requested shifts with the following remarks:
"*The experiment proposes an extension of the emission channelling measurements with position-sensitive detectors to isotopes with shorter half lives and lower electron energies. The technique is routinely used at ISOLDE for studies of lattice location of radioactive impurities implanted into single crystals, and can now be broadened due to the availability of new on-line emission channelling setups and faster readout systems. The Committee judged the proposal as a step forward in terms of instrumentation, and considered the first planned applications of interest. Special emphasis was given to the investigation of lattice location of transition metals in semiconductors, for which emission channelling is ideally suited, in order to understand the physics of ferromagnetism in semiconductors. The application of Auger electrons for channelling studies is very demanding and its advantages are not obvious, hence the feasibilityof this technique should be demonstrated first with a selected case. With this provision, the Committee decided to recommend for approval by the Research Board a total of 20 shifts for a 2 year period, after which the proponents should report on the achievements with special emphasis on the online measurements. The possible technological applications of this project were underlined.*"

The 20 shifts granted have meanwhile been used and as requested we are herewith reporting on the status of IS453, followed by a request for 22 additonal shifts in order to continue the experimental studies.

The aims of the presented addendum are as follows:
- continue the successful work in the fundamentally and technologically relevant field of the lattice location of transition metals and dopants in semiconductors using as probes the isotopes ^{56}Mn (1.6 h), ^{59}Fe (45 d), ^{61}Co (1.6 h), ^{65}Ni (2.5 h);
- following a short but successful run with ^{27}Mg (9.46 min), perform detailed lattice location studies of th Mg acceptor in nitride semiconductors;
- re-establish lattice location studies of the short-lived *alpha* emitter ^{8}Li (838 ms) with experiments on the lattice location of Li in ZnO.

2) Status report IS453 EC-SLI

In the time period May 2007 - October 2009 IS453 had the following GPS beam times scheduled

- ZrO hot plasma run May 2007 (^{73}As), this run had to be canceled due to ISOLDE problems
- UC_2-W RILIS Mn run June 2007 (^{56}Mn, ^{61}Mn, ^{59}Mn→^{59}Fe, ^{61}Mn→^{61}Fe→^{61}Co) LA2 beamline
- ZrO hot plasma run May 2008 (^{73}As) SSP collection chamber
- UC_2-W RILIS Mn run Sept. 2008 (^{56}Mn, ^{61}Mn, ^{59}Mn→^{59}Fe, ^{61}Mn→^{61}Fe→^{61}Co, ^{111}In) LA2 beamline

- ZrO hot plasma run May 2009 (^{73}As) SSP collection chamber
- UC_2-W RILIS Mn run July 2009 (^{56}Mn, ^{61}Mn, ^{59}Mn$\rightarrow^{59}$Fe, ^{61}Mn$\rightarrow^{61}$Fe$\rightarrow^{61}$Co) LA1 beamline
- SiC-W RILIS Mg run August 2009 (^{27}Mg) GHM beamline.

The UC_2-W RILIS Mn runs of June 2007 and Sept. 2008 were quite successful, allowing us to perform on-line lattice location experiments of ^{56}Mn and ^{61}Co in both GaN and ZnO, as well as of ^{56}Mn in GaAs and Ge. The Mn run of July 2009, on the other hand, suffered from the fact that a few hours into the run the GPS high voltage became unstable so that only 30 kV could be used. This made it necessary to redo stable beam tuning at the lower acceleration energy, causing considerable delay and loss of beam time. As a result of the delay and due to the poor beam collimation at 30 kV and very poor radioactive yields (a factor of ~50 times lower than in the previous two years) we were only able to do preliminary studies of the lattice location of ^{56}Mn in two Si samples. The experiments hence suffered from very low count rates and excessive background resulting from activity deposited on the collimator. During all of the three Mn runs, off-line collections of ^{59}Mn$\rightarrow^{59}$Fe were done in a variety of samples, including Ge, ZnO, GaN, GaAs, $SrTiO_3$, and $KTaO_3$, which were measured during the subsequent months using our three off-line setups available for that purpose.

The positions at the LA2 or LA1 beam lines, which were used during the three on-line Mn runs turned out to be far from optimum. At the end of these relatively long beam lines the focusing of the ISOLDE beam is already quite poor, resulting in only modest transmission of around 20-30% through the 1 mm collimator required for the emission channeling experiments. In August 2009 we could move our setup to a permanent position at the GHM beamline, located only a few meters from the switchyard, thus allowing us to achieve transmissions up to 50%.

During the SiC-W RILIS Mg run in September 2009 we managed to do first lattice location experiments with ^{27}Mg (9.46 min) implanted into GaN and AlN. The biggest problem to be overcome was the ~1.5 nA stable ^{27}Al contamination that was present at mass 27 in this type of target. However, the ^{27}Al contamination could be significantly reduced by running the SiC target and line at very low temperatures (estimated target temperature 1000-1200°C). For that purpose the target and line heating currents were reduced to 220 A and 250 A, respectively, so that the proton beam represented the main source of target heating. Under these conditions the ^{27}Al current could be reduced to 2-4 pA, with around 0.3 pA of ^{27}Mg left. In radioactive equilibrium this ^{27}Mg current results in around 1500 Hz count rate in our position-sensitive detector, enough to carry out on-line measurements during implantation but not sufficient to perform thermal annealing of the sample (a typical annealing step requires ~20 min waiting time before the measurement can start). In order to deal with this situation better next year, the Si pad detector will be equipped with an aluminated Mylar foil window, allowing to operate the detector while the sample is heated and thus allowing to measure directly at higher implantation temperatures. Moreover, according to information presented by Th. Stora at the ISOLDE 2009 Workshop, it should be possible to decrease the ^{27}Al contamination further by using highly pure SiC as target material, a neutron converter and the ^{30}Si(n,α)^{27}Mg reaction to produce ^{27}Mg, and GdB_6 cavities in the ionizer.

The As beam times were used in order to produce samples for testing the performance of the Si pad detector system with low energy electrons from ^{73}As, for testing the response of the MediPix and TimePix detectors and to perform lattice location studies of this isotope in GaN, ZnO and Ge. Unfortunately the 1 mm thick pad detector used could not be fully depleted without the severe risk of micro-breakthroughs that overload and may damage single pixels. The detector hence has to be used with the pad side facing the sample. While this does not degrade its performance as a fast position-sensitive detection system for high-energy β particles, the relatively thick entrance window on the pad side causes considerable energy loss and energy straggling. While 50 keV conversion electrons were still detected with an energy resolution around 3.5 keV and a deposited energy of 45 keV, it was not feasible to detect electrons below 35 keV with sufficient energy resolution.

The tests of the Goettingen CCD detector for extremely low-energy electrons (conversion or Auger electrons below 40 keV) mentioned in our original proposal had to be postponed since the device broke down in 2007 and had to be sent for repair to the manufacturer. Once reliable operation of the

CCD can be guaranteed, we will resume these tests. In the meantime we will concentrate on the lattice location experiments with short-lived β^- emitters and on further testing and optimization of the newly acquired TimePix detectors (cf. sect. 4).

Following its commissioning in 2007, in 2008 the ITN/CFNUL on-line emission channeling chamber was upgraded with a closed-cycle He refrigerator which allows cooling the sample to 50 K. ITN has recently bought a high-precision 3-axis goniometer with *x*,*y*,*z* translation stage from the company Panmure which will be commissioned during the following months.

The publications of our group since the year 2006 are listed in section 6 as those resulting still from the previous experiment IS368 "Lattice location of transition metals and rare earths in semiconductors" [1-8] and those that incorporate already data taken as part of the current experiment IS453 EC-SLI [9-14].

The following three PhD students are currently working within the scope of IS453

Lino Pereira (since Jan. 2008), topic of thesis: "Diluted magnetic wide-gap semiconductor nanostructures produced by ion implantation", Joint PhD between Universidade do Porto, Portugal, and Katholieke Universiteit Leuven, Belgium.

Matthias Nagl (since July 2008): thesis on Digital PAC and emission channeling experiments at ISOLDE/CERN, Universität Göttingen.

Lígia Amorim (since Jan. 2010), topic of thesis: "Lattice location studies of implanted Mg in the III-nitride semiconductors GaN, AlN and InN", Instituut voor Kern- en Stralingsfysica, Katholieke Universiteit Leuven, Belgium.

The following three PhD students have finished their thesis within our group since 2006:

Elisabete M.C. Correia Rita, topic of thesis: "Properties and local environment of *p*-type dopants and photoluminescent rare earths implanted into ZnO single crystals", Universidade de Lisboa, 13.10.2008.

Stefan Decoster, topic of thesis: "Ion implantation in Ge: Structural and electrical investigation of the induced lattice damage & study of the lattice location of implanted impurities", Instituut voor Kern en Sterrenkunde, Katholieke Universiteit Leuven, 17.6.2009.

Ana Claudia Lourenço Santana Marques, topic of thesis: "Advanced Si pad detector development and $SrTiO_3$ studies by emission channeling and hyperfine interaction experiments", Universidade de Lisboa, 15.10.2009.

3) Physics case

The ability to use short-lived isotopes in emission channeling has provided many opportunities for doing lattice location studies of elements that were previously not feasible to use for that purpose at ISOLDE, in particular Mn, Co, Ni and Mg. In the following we will describe the progress in the initial fields of work outlined in the original proposal, followed by proposed future activities.

3a) Lattice location of transition metals in semiconductors

Our lattice location studies of transition metals (TMs) in semiconductors are motivated by the widespread interest in these systems due to the following two topics:

- the role of TMs as magnetic impurities in semiconductors, in particular the quest for room temperature ferromagnetic semiconductors which is an important feature for the realization of future spintronic devices; the systems which are of interest in that respect are the TM-doped wide bandgap semiconductors ZnO and GaN but also the "classical" dilute magnetic semiconductor $Ga_{1-x}Mn_xAs$;
- the role of TMs as major contaminants in Si and Ge, where they are responsible for the creation of deep levels and act as "lifetime killers" for minority carriers.

Within the scope of IS453 we have initially focused our lattice location experiments on Co and Mn in the wurtzite semiconductors ZnO and GaN. As was mentioned already, TM doping of these has been

reported to result in room temperature ferromagnetism [16-22]. However, the nature of the ferromagnetism is still controversial, related to the fact that the microstructure of the TMs in these materials is poorly understood. In particular it is heavily debated whether the ferromagnetism results from isolated TM impurities in the sample, as in an ideal dilute magnetic semiconductor, or is due to the formation of TM clusters or even second phases of ferromagnetic compounds in the sample. With respect to TM-doped ZnO, Ref. [21] points out that "*In particular, methods such as EXAFS or XPS that establish lattice location or chemical state should be applied in order to give more insight into possible mechanisms for the observed ferromagnetism.*" It is hence clear that emission channeling experiments which directly establish the lattice site of the TMs will provide valuable information. On the other hand, recent results of the ^{57}Fe Moessbauer experiment IS443 at ISOLDE have established that dilute ^{57}Fe in ZnO is only subject to paramagnetism. With respect to TM-doped GaN, Ref. [22] remarks that "…*it has been found that spinodal decomposition into regions more and less rich in magnetic ions appears to be a general result of the integration of TM and rare earths into a semiconductor matrix.*". While emission channeling is not directly sensitive to the nature of the atoms surrounding the probe atom, it can indirectly provide information on the formation of disordered clusters. Probe atoms incorporated in disordered clusters would lead to the occupation of low-symmetry sites, which would cause a loss of anisotropy and hence show up as an increase of the random fraction in the analysis.

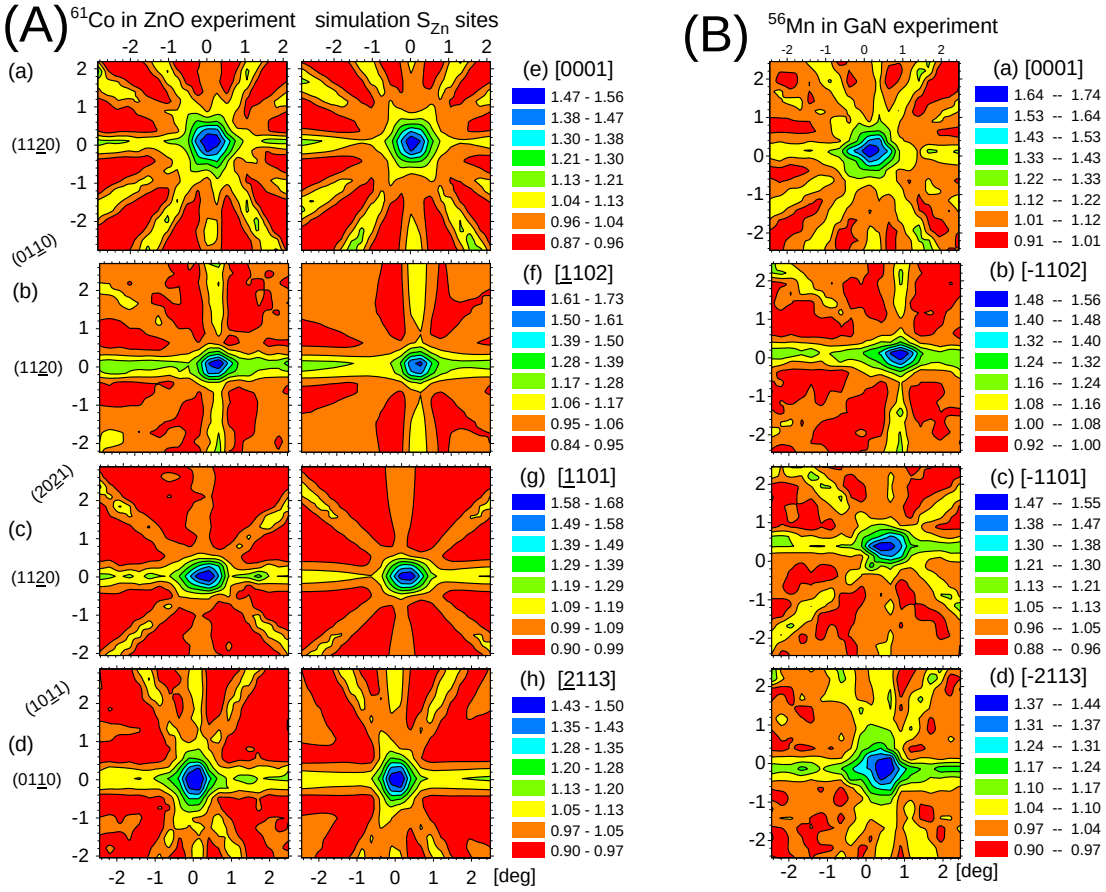

Figure 1: (A) Experimental and simulated β^- emission channeling patterns from ^{61}Co in ZnO following annealing at 800°C. The left column are experimental results along the four major axial directions [0001], [-1102], [-1101] and [-2113], while the columns to the right show the best fit of simulated emission patterns to the experimental data, taking into account only contributions from 100% of ^{61}Co emitter atoms on substitutional Zn sites S_{Zn}.
(B) Experimental β^- emission channeling patterns from ^{56}Mn in GaN following annealing at 900°C.

Figure 1 (A) shows the emission channeling patterns obtained from the probe atom ^{61}Co incorporated into ZnO following annealing at 800°C. Since pure radioactive beams of Co ions are not available at

ISOLDE, the precursor isotope ^{61}Mn (0.71 s) was implanted from a UC_2 Mn RILIS target. ^{61}Mn decays into ^{61}Fe (6 min) and then further into ^{61}Co (1.6 h) and both decays are accompanied by large nuclear recoils (around 520 and 103 eV) so that the daughter atoms are recoil-implanted. The patterns were recorded after waiting at least 30 min following the end of the implantation, thus assuring that only ^{61}Co probe atoms contribute to the channeling patterns. Towards the end of the waiting time the sample was annealed for 10 min at 800°C and cooled back to room temperature. The comparison of measured and best-fit patterns shows that ~100% of ^{61}Co probe atoms occupied the substitutional Zn sites. Similar patterns were measured already in the as-implanted state. The emission channeling technique hence shows that following low-fluence (~3×10^{12} cm^{-2}) implantation of Co and annealing at 800°C no disordered Co clusters are formed. If the formation of clusters should have occurred in this experiment, it could only be in such a way that the Co atoms are still occupying sites which are perfectly aligned with the Zn atomic rows in the sample, in other words equivalent to ideal S_{Zn} sites. In a similar experiment, ^{56}Mn (1.5 h) was implanted into a GaN sample. The β^- emission channeling patterns measured during its decay, following annealing at 900°C, are shown in Fig. 1(B). While the full analysis of this experiment is currently under way, the similarity of the patterns to those measured for ^{61}Co in ZnO shows that ^{56}Mn clearly prefers to occupy substitutional Ga sites S_{Ga}. Similar results were obtained from ^{56}Mn in ZnO and ^{61}Co in GaN, showing that both of these two transition metals have a clear preference for substitutional cation sites in the two wurtzite semiconductors.

In the original proposal we had also proposed investigating samples preimplanted with stable transition metal isotopes in order to study the lattice site as a function of TM concentration. However, due to the poor reproducibility of the 60 kV operation at ISOLDE, it was impossible to guarantee a match between the energy chosen in the preimplantation and the acceleration energy that had to be used during the radioactive run. We therefore have decided to give up doing this kind of experiments.

Except for the case of ^{65}Ni, the studies of TMs in ZnO and GaN have been realized to a large extent, and in the future we want to focus on the case of the narrow band gap materials Si, Ge and GaAs, as will be described in the following.

The ternary compound $Ga_{1-x}Mn_xAs$ represents the best understood [23] dilute magnetic semiconductor system which exhibits ferromagnetism, although the maximum Curie temperature T_C realized (around 200 K) is currently still considerably below room temperature. Whereas it is well-known that T_C in $Ga_{1-x}Mn_xAs$ increases with increasing Mn fraction x and that Mn mostly replaces Ga atoms, it was suggested that there is a limit imposed by the amount of Mn atoms that are incorporated interstitially [24-26]. However, the exact location of interstitial Mn is unknown and its thermal stability and diffusion behaviour are uncertain as well. In addition to ZnO and GaN we have therefore also studied the lattice location of ^{56}Mn implanted into p^+-GaAs:Zn. As a representative example, Fig. 2 compares the normalized experimental ^{56}Mn β^- emission yields along the four major directions, measured after 300°C annealing, with the best fits of theoretical patterns, which were obtained for a combination of 70% of ^{56}Mn on substitutional S_{Ga} and 30% on tetrahedral interstitial T_{As} sites.

The fraction of ^{56}Mn on S_{Ga} and T_{As} interstitial sites as a function of annealing temperature is shown in Fig. 3. As is easily visible, the interstitial Mn fraction only starts to disappear for annealing temperatures above 400°C, which is contrary to the claim reported in the literature that Mn interstitials anneal by means of outdiffusion from the sample at temperatures around 200°C [24-26]. However, it cannot be excluded that the behaviour of Mn is different in real $Ga_{1-x}Mn_xAs$ samples, where the Mn fraction x is typically around 1-5%, although emission channeling experiments in p^+-GaAs should most closely resemble the situation in $Ga_{1-x}Mn_xAs$ samples, which are typically also highly p-type due to the acceptor character of Mn_{Ga}. On the other hand, we have preliminary data that shows that the lattice location of ^{59}Fe in GaAs depends on the doping character of the material. We have previously observed similar effects for Cu in Si [1, 27-28] and the most likely explanation is due to the fact that the charge state of the impurity and other defects in the sample may change with the position of the Fermi level. While the results of the emission channeling experiment in p^+-GaAs are currently being prepared for publication, in the future we intend to systematically study the lattice location of ^{56}Mn and ^{59}Fe in GaAs also in semi-insulating *si*-GaAs and highly *n*-doped n^+-GaAs, and, if we can establish a collaboration with a group growing such material, in actual $Ga_{1-x}Mn_xAs$ samples.

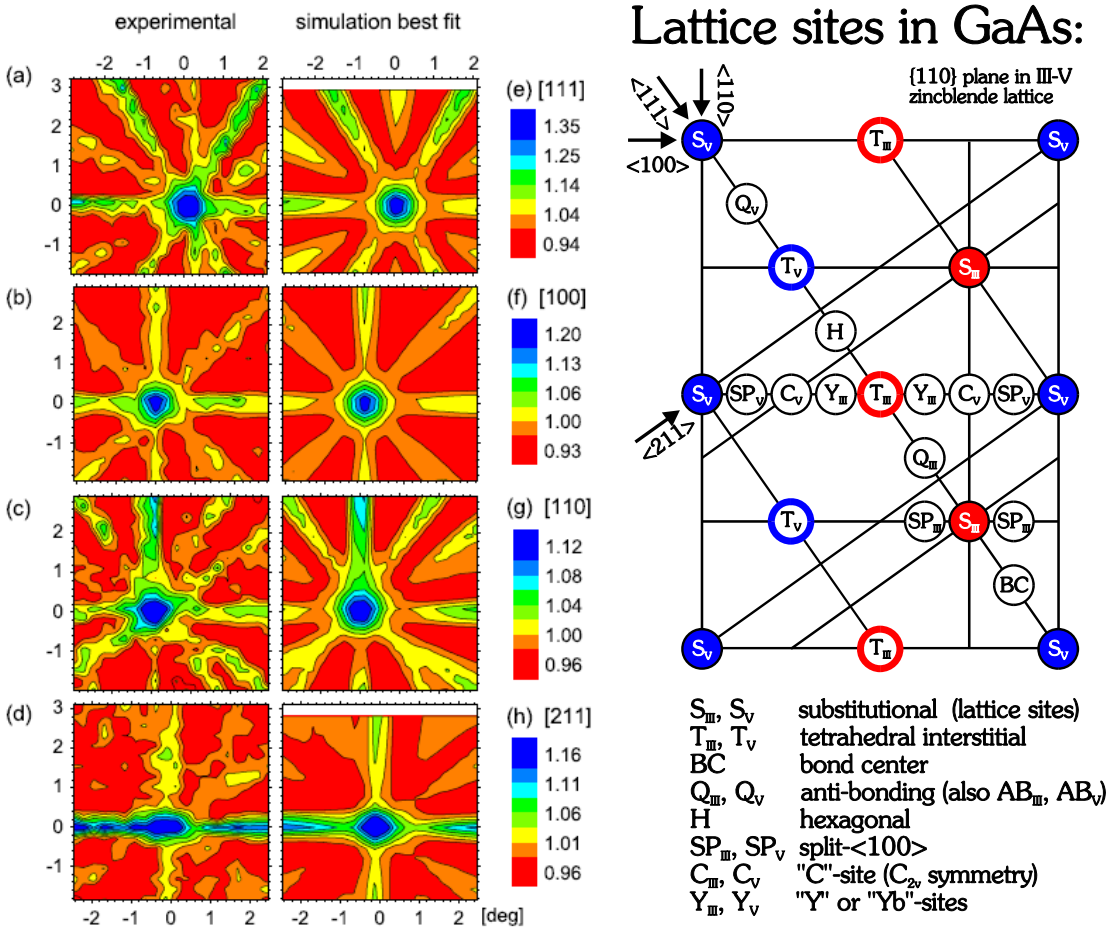

Figure 2: Left: Experimental and simulated β^- emission channeling patterns from ^{56}Mn in p^+-GaAs:Zn following annealing at 300°C. The left column are experimental results along the four major axial directions [111], [100], [110] and [211], while the columns to the right show the best fit of simulated emission patterns to the experimental data, taking into account only contributions from ^{56}Mn emitter atoms on substitutional Ga sites S_{Ga} (~70%) and interstitial T_{As} sites (~30%).
Right: Illustration of high-symmetric sites in GaAs. Manganese occupies the substitutional Ga sites S_{Ga} (shown as S_{III} in the figure) and the interstitial T_{As} sites (shown as T_V).

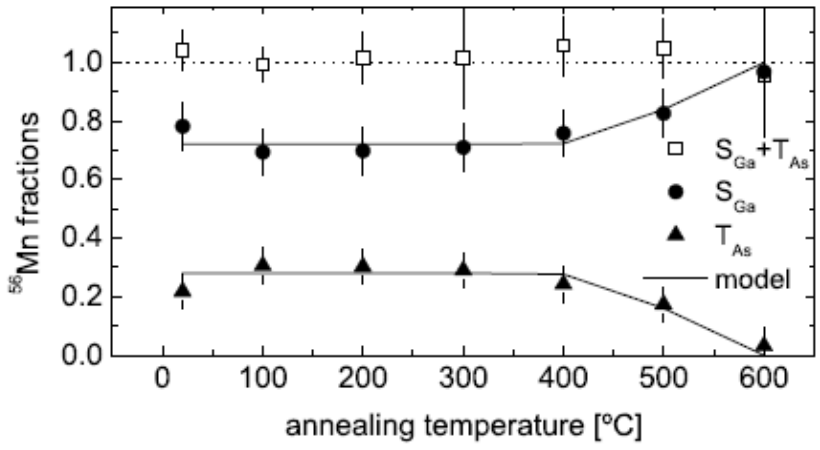

Figure 3: Fractions of ^{56}Mn on S_{Ga} and T_{As} sites derived from the fits to the experimental patterns as a function of annealing temperature. Open squares: total $S_{Ga} + T_{As}$ fraction derived from the [111],

[100], [110] and [211] fits; solid circles: relative fractions of S_{Ga} and (solid triangles) relative fractions of T_{As} averaged from the [110] and [211] fits.
As was mentioned above, the main interest in TMs in Si arises from the fact that these elements are unavoidable contaminants in the Si production process and must be carefully controlled during wafer processing. This issue is well known in the microelectronics and photovoltaics industry, where sophisticated TM gettering procedures are integrated into the device production [29-31], explaining the continued interest in the behaviour of TMs in Si throughout the last 6 decades. This is even further exemplified due to the need of the photovoltaic industry to consider the use of so-called "metallurgical grade" Si [32]. While this unrefined starting material has a 30-100 lower cost than "semiconductor grade" Si, it suffers from metal contaminations in the % range. Finding cheap and efficient ways to reduce the metal contamination without the need of an additional gas-phase purification process, or to limit the TM impact on device performance is considered as a route for significantly reducing the price of Si solar cells.

Within the scope of our previous experiment IS368 we have addressed the basic properties of TMs in Si by studying the lattice sites of ^{59}Fe and ^{67}Cu as a function of annealing temperature in Si samples of different doping types. It is now intended to continue these studies with ^{56}Mn and ^{65}Ni, possibly also ^{61}Co. First emission channeling results obtained from ^{56}Mn in Si indicate that substitutional Mn is dominant, while interstitial Mn cannot be quenched in large amounts, i.e. surprisingly the behaviour of Mn in Si is more similar to Cu [1, 27-28] than it is to Fe [1, 33]. Lattice location experiments of ^{56}Mn offer the opportunity to compare the channeling data to the results of ^{57}Mn→^{57}Fe Moessbauer experiments (cf. proposal presented to the INTC.)

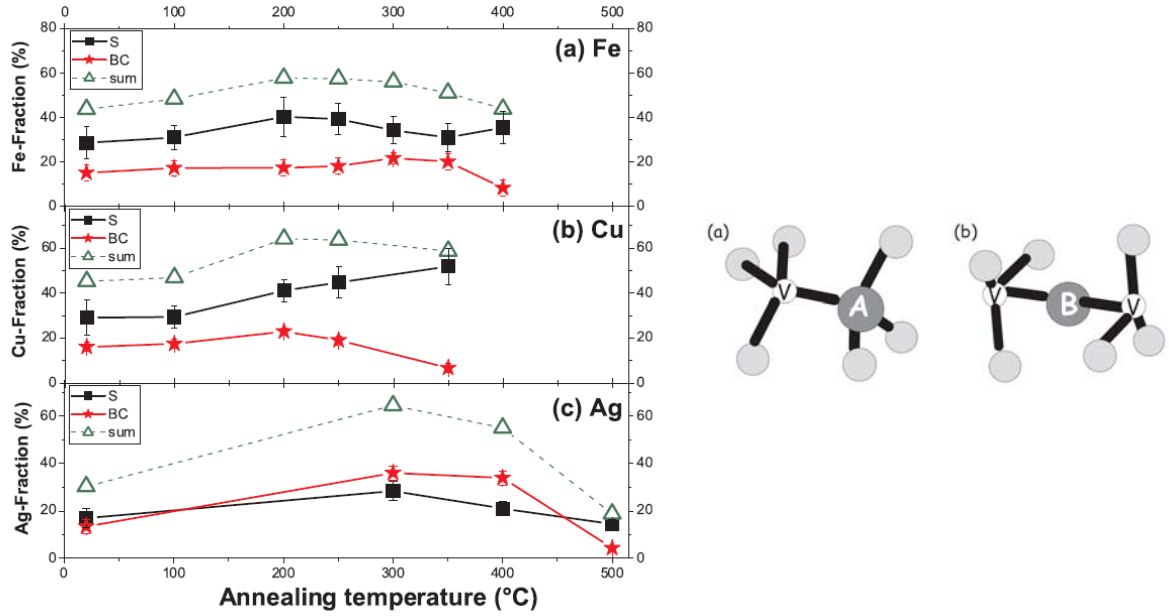

Figure 4: Left: Fractions of implanted ^{59}Fe, ^{67}Cu and ^{111}Ag on substitutional S and bond-centered (BC) sites in Ge as a function of annealing temperature.
Right: The two competing structures for TM-*V* complexes in Ge: the "full vacancy" configuration (a) consists of a TM atom "A" which remains substitutional after trapping another vacancy *V*; in the "split vacancy" configuration (b) the TM atom "B" moves from its substitutional site until it is located at the BC position centered between the two adjoining vacancies *V*. While the substitutional fraction in the three experiments is interpreted as isolated substitutional TMs that do not have trapped a vacancy (not shown), the BC position is identified with complexes of type "B".

Recently, Ge is being considered as an important future material to replace Si in some semiconductor applications. When compared to silicon, Ge has a higher free carrier mobility and a lower dopant ionization temperature, which would allow to realize faster integrated circuits. Since it is known that TMs also form deep levels in Ge and hence play a similar role as in Si, the corresponding issues with

respect to Ge processing are currently addressed by many research groups [34]. We have previously performed lattice locations studies of ^{67}Cu and ^{111}Ag within the scope of IS368 and recently also of ^{59}Fe within IS453 in Ge [10]. Our main finding was that apart from substitutional sites the TMs Fe, Cu and Ag are also found on the so-called bond-center (BC) sites (Fig. 4). By means of investigating the formation energy of various TM defects with the help of density functional theory, the bond-centered location was identified as TM atoms inside a double vacancy. Rather than remaining on its substitutional position, this so-called split-vacancy configuration is formed when a substitutional TM traps a Ge vacancy (Fig. 4 right). We intend to continue the lattice location experiments of TMs in Ge by studying the lattice location of the now available short-lived TM probes as well.

Proposed experiments: We propose to continue the studies of the lattice location of TMs in Si, Ge and GaAs by means of β^- emission channeling using the short-lived isotopes ^{56}Mn ($t_{1/2}$=1.5 h), ^{61}Co ($t_{1/2}$=1.6 h), and ^{65}Ni ($t_{1/2}$=2.5 h). Apart from studying intrinsic crystals, in particular the use of n^+ and p^+ materials will reveal the influence of the type of doping on the lattice position, an effect that was observed previously for Fe and Cu in Si. While ^{56}Mn and ^{65}Ni can be directly obtained at ISOLDE from RILIS laser ion sources, ^{61}Co is to be implanted via the precursor isotope ^{61}Mn, exploiting the decay chain ^{61}Mn (0.71 s) $\rightarrow$ ^{61}Fe (6 min) $\rightarrow$ ^{61}Co. With respect to ZnO and GaN, the measurements with ^{65}Ni are still missing. As previously, a few shifts of the isotope ^{59}Mn (4.6 s) $\rightarrow$ ^{59}Fe (45 d) are also requested in order to continue lattice location studies of ^{59}Fe, mostly in GaAs. Our emission channeling experiments on Mn and Fe in Si will benefit from a direct comparison to the results of ^{57}Mn$\rightarrow$^{57}Fe Moessbauer studies (the successor to experiment IS443, a proposal to be assessed during the same INTC meeting).

3b) Lattice location of Mg in nitride semiconductors

As stated already in our original proposal, Mg is the only feasible *p*-type dopant in the technologically important wide band gap semiconductor GaN [35-36]. All applications of GaN involving *pn*-junctions such as blue or white LEDs, blue lasers and high-frequency power diodes rely on Mg doping. In electrical studies it was found that typically ~1% or less of Mg in GaN contributes holes to the valence band while from its energy level of ~160 meV above the valence band one would expect around 10% of ionized acceptors [35]. It is unknown whether the large electrically inactive fraction is caused by Mg occupying other lattice positions than substitutional Ga (e.g. interstitial sites), the formation of complexes between Mg and other defects, e.g. Mg-H or Mg-V_N complexes, or by other effects [36]. Moreover, recent experimental evidence [37] indicates that there exist two types of Mg acceptor centers in GaN, although no detailed structural models have been proposed in order to distinguish them.

Our recently performed emission channeling measurements of ^{27}Mg in GaN and AlN were the first direct lattice location experiments of Mg in nitride semiconductors. They showed that the large majority of ion implanted Mg is incorporated on Ga or Al sites. Preliminary analysis of the GaN data by .means of fitting the experimental patterns to the results of simulations for ^{27}Mg on different lattice sites (cf. Fig. 4) gave no indication for large fractions of Mg on interstitial sites. However, we could not yet determine the influence of post-implantation annealing on the lattice site occupancy of Mg. Since a typical annealing procedure lasts about 20 min, relatively high fluences of short-lived ^{27}Mg (9.5 min) would be required in order to compensate for the decay losses during thermal processing. With the low Mg beam intensities resulting from the relatively cool target operating conditions required in order to suppress the Al contamination, this seems impossible. In order to circumvent this problem, we will equip the detector with a shielding of thin aluminated Mylar foil, which will considerably reduce its exposure to light and thermal radiation during sample heating, without distorting the position resolution. In previous experiments this allowed to perform emission channeling lattice location measurements in-situ during sample annealing at 900°C [38-39].

While Mg-doping of GaN is technologically well established and, to a lesser extent, also Mg doping of AlN, very little is known on the behaviour of Mg in InN [40]. Single-crystalline InN layers of good quality are only available since a few years. It is theoretically predicted [41] that Mg should be most stable on substitutional In sites and constitute a promising acceptor, although it may be subject to form

stable pairs with nitrogen vacancies V_N. Clearly lattice location experiments of Mg in InN will be relevant.

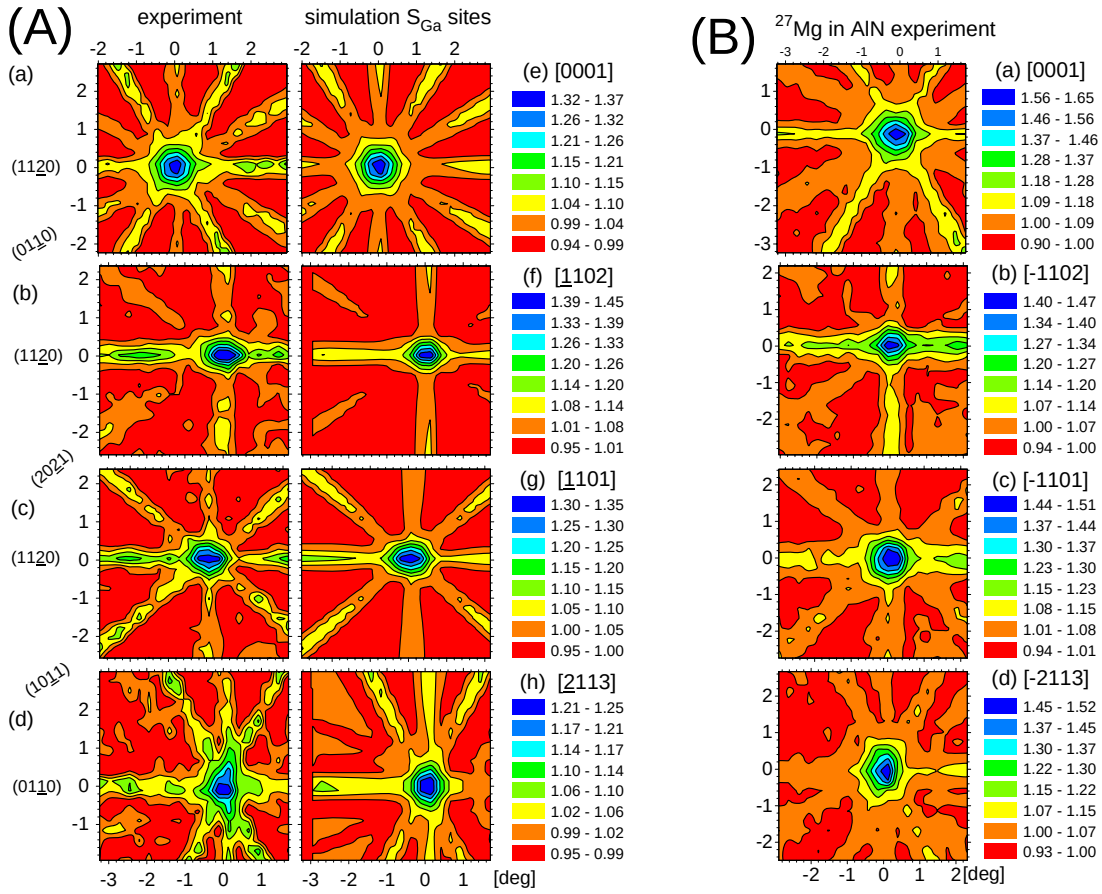

Figure 5: (A) Experimental and simulated β^- emission channeling patterns from ^{27}Mg in GaN in the as-implanted state. The left column are experimental results along the four major axial directions [0001], [-1102], [-1101] and [-2113], while the columns to the right show the best fit of simulated emission patterns to the experimental data, taking into account only contributions from ^{27}Mg on substitutional Ga sites S_{Ga}.
(B) Experimental β^- emission channeling patterns from ^{27}Mg in AlN in the as-implanted state.

Proposed experiments: We propose to continue and intensify the study of the lattice sites of implanted short-lived ^{27}Mg ($t_{1/2}$=9.46 min) in the nitride semiconductors GaN and AlN, in particular at high temperatures. Instead of implanting at room temperature and performing annealing steps, we intend to implant and measure in-situ at high temperatures. In addition, the lattice sites of ^{27}Mg in InN will be investigated for the first time. While the experiments are already feasible with the currently achieved level of ^{27}Al contamination, we ask to consider reducing it further by using highly pure SiC as target material, a neutron converter and the ^{30}Si(n,α)^{27}Mg reaction to produce ^{27}Mg, and GdB_6 cavities in the ionizer. Which of these measures are to be adopted should be decided by the ISOLDE technical group, taking into account the related workload and chances of success.

3c) Lattice location of Li in ZnO

ZnO is a technologically promising II-VI semiconductor which has the same hexagonal wurtzite structure and similar properties to GaN. As was the case in the beginning of GaN technology, reproducible *p*-type doping is now a major obstacle in the way of realizing ZnO based devices. The alkali metal Li is one of the impurities which are currently discussed as possible *p*-type dopants in ZnO [42-45]. As early as 1960 it had already been proposed that Li exists in ZnO not only as substitutional acceptor impurity on Zn sites but also in an interstitial donor form, and that Li would

therefore be an amphoteric impurity [46]. Although this hypothesis was recently backed up by theoretical considerations [Lee 04, Wardle 05], to date no direct experimental proof of the existence of interstitial Li in ZnO is available, neither is it known which of the various interstitial sites in the wurtzite structure (T, H, O, AB etc. sites) is possibly preferred by Li.

In the past, the lattice location of Li has been successfully studied at ISOLDE by means of α emission channeling of ^{8}Li ($t_{1/2}$=838 ms) in a variety of semiconductors including Si, Ge, diamond, SiC, GaAs, AlGaAs, GaP, InP, InSb, ZnSe, ZnTe, CdTe, AlN and GaN (see e.g. [15, 47-48]). One of the major conclusions drawn from these studies was that Li which is implanted at low temperatures occupies preferentially interstitial lattice sites, but in a certain transition temperature regime, related to the onset of interstitial Li diffusion, changes its lattice site. Remarkably, in all investigated III-V and II-VI compound semiconductors a site change from interstitial sites to substitutional cation sites was observed. In the case of GaN, which has very similar properties to ZnO, the site change occurred around 700 K [48], and we hence predict a similar behaviour for Li in ZnO. Our proposed emission channeling experiments of ^{8}Li in ZnO are therefore expected to unambiguously establish the existence of both interstitial Li_i and substitutional Li_{Zn} in ZnO, identify the preferred interstitial site of Li_i, and also yield valuable results on its diffusion behaviour.

Proposed experiments: We propose to study the lattice sites of implanted ^{8}Li (838 ms) in ZnO by means of α emission channeling as a function of implantation temperature between 50 K and 800 K. This experiment will make use of the on-line setup and the position-sensitive α detection system from the University of Göttingen, as previously used in IS342.

4) Test isotopes for detector development

In 2007 we established an agreement with the CERN MediPix collaboration, which is supplying us with advanced Si pixel detectors of the TimePix type in order to test their application in electron emission channeling. The detectors consist of 1.5×1.5 cm^2 sized segments of 256×256 pixels with a pixel size of 55 μm, which are directly bonded on their backside to preamplifier chips. Figure 6 shows a Quad TimePix detector consisting of 4 segments (total active area 3×3 cm^2, 512×512 pixels).

While the MediPix detectors only allow for setting a lower level threshold which triggers the readout of a pixel but does not provide a proper energy signal, the TimePix detectors achieve real energy resolution by means of measuring the pulse length above threshold following particle impact, the so-called time-over-threshold (TOT) method. First tests with a weak ^{73}As source mounted directly in front of the detector showed that an energy resolution around 6.5 keV can be achieved using the TOT method, a value that is only about a factor of 2 worse than for our Si pad detectors.

During summer 2009 a Quad TimePix detector, which was fully mounted at the EC-SLI online setup has been tested for the first time using the β^- particles emitted by ^{89}Sr (50 d) implanted into $SrTiO_3$. The resulting emission channeling patterns are shown in Fig. 7 and prove that the detectors which were initially developed for position sensitive detection of X-rays in medical diagnostics in air, also work for the detection of high-energy electrons inside vacuum. Our aim is to continue characterizing and optimizing the response of the detectors and readout electronics for conversion electrons and β particles so that they can be used in the future for emission channeling experiments that would benefit from the increased position resolution due to the small pixel size of 55 μm. While the position resolution of the patterns in Fig. 7 is entirely limited by the 1 mm diameter beamspot size on the sample, using smaller beamspots would in principle allow to decrease the position resolution to values which are limited either by the pixel size of the detector or the straggling of the electrons when being stopped in the detector. However, the implanted beamspots cannot be made arbitrarily small, certainly not much less than 0.5 mm. Since the detector was mounted at a distance of 30 cm from the sample, the angular resolution resulting from the geometry alone is estimated to be ~0.05° standard deviation. The patterns in Fig. 7 have somewhat worse resolution since the β^- particles from ^{89}Sr (mean β^- energy 586 keV) will typically straggle around 1 mm in the detector, and hence hit a number of pixels.

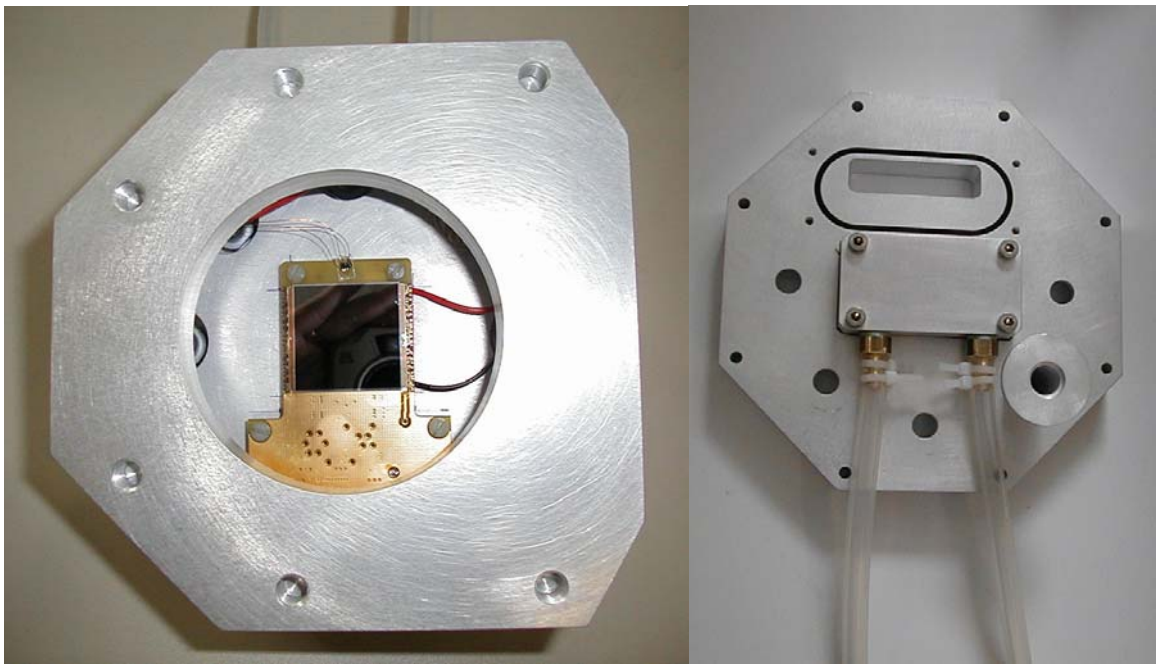

Figure 6: Left: A Quad TimePix detector mounted inside a housing which can be directly attached to the EC-SLI on-line setup. On top of the printed circuit board a Si diode is visible which measures the temperature and provides a control signal for the Peltier elements that provide active cooling of the detector and dissipate the heat created by the preamplifiers.
Right: Outside back view of the detector flange before mounting the electrical signal feedthroughs. In the center the cooling water circuit is visible which provides the heat sink for the Peltier elements inside the vacuum.

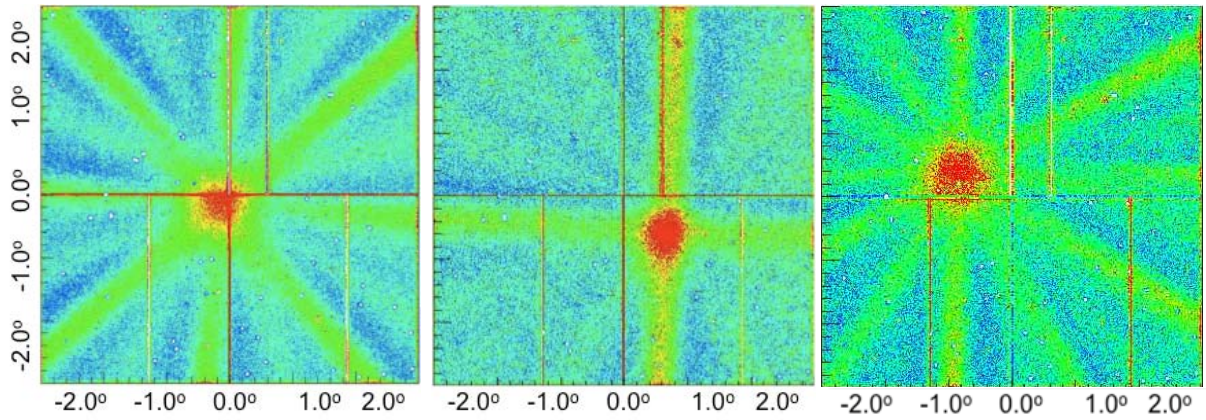

Figure 7: β^- emission channeling patterns from ^{89}Sr implanted into $SrTiO_3$ (located on substitutional Sr sites) measured around the <100> (left), <211> (center) and <111> (right) directions using a prototype Quad TimePix detector with 512×512 pixels. The straight red or green lines visible in the patterns arise due to the higher count rate in the pixels at the boundary areas of the four separate detector segments, which are 3 times the size of the interior pixels. This effect has not yet been corrected in the patterns. However, three other lines of enhanced count rate are also visible, indicating problems due to excessive noise in some areas of this prototype detector.

Proposed experiments: Two shifts of ^{73}As and ^{75}Se are requested in order to produce long-lived samples that can be used to test and optimize the performance of the TimePix detectors under realistic conditions. The isotopes ^{73}As and ^{75}Se are particularly suitable for such tests since they provide sets of conversion electron lines at low and intermediate energies (12, 42 and 52 keV for ^{73}As, 9 separate

lines in the energy range 85-293 keV for ^{75}Se) and possess conveniently long half lives of 80.3 d and 120 d. We know from previous experiments that both isotopes can be implanted in ZnO samples at relatively high fluences (10^{15} cm^{-2}) and still result in excellent channeling effects due to the incorporation of ^{73}As on Zn and ^{75}Se on O sites. An added advantage is that they are both available at ISOLDE with high yields from the same type of targets, either ZrO_2 or Nb foil.

5) Beam request and experimental requirements

We request a total of 22 shifts over a period of 2-3 years for the isotopes specified below in Table 1. All requested targets are standard ISOLDE targets and the beam times can hence be shared with other users. While the yields of most isotopes are known from previous runs, the yield of ^{65}Ni has been taken from the ISOLDE yield data base, updated by information from Th. Stora and U. Köster.

isotope	number of shifts	target	ion source	minimum yield [atoms/s/μA]
^{56}Mn (1.5 h)	3	UC_2-W	RILIS Mn	5×10^7
^{61}Mn (4.6 s) $\rightarrow$ ^{61}Fe (6 min) $\rightarrow$ ^{61}Co (1.6 h)	1	UC_2-W	RILIS Mn	2×10^6
^{59}Mn (0.71 s) $\rightarrow$ ^{59}Fe (45 d)	1	UC_2-W	RILIS Mn	10^8
^{65}Ni (2.5 h)	4	UC_2-W	RILIS Ni	5×10^7
^{27}Mg (9.5 min)	8	UC_2-W	RILIS Mg	5×10^6
^{8}Li (838 ms)	3	Ta	W surface ioniz.	10^7
^{73}As (80 d) ^{75}Se (120 d)	2	ZrO_2 or Nb foil	Hot Plasma	10^8
	Total: 22			

Table 1: Requested isotopes, number of shifts, targets, ion sources and minimum yield requirements.

As was mentioned already, the EC-SLI on-line emission channeling setup is currently installed at the GHM beamline of GPS. Since this position provides much more favourable conditions with respect to the required collimation of the ISOLDE beam than the LA1 or LA2 beam lines used previously, we ask to keep this position.

For implantation of the longer-lived isotopes, we will use the standard solid-state physics implantation chamber and the corresponding measurements will be done off-line with the emission channeling setups located in the ISOLDE laboratory for radioactive off-line experiments in building 275.

6) Publications and References

6a) Publications since 2006 related to our previous experiment IS368 (and not yet reported in the 2006 EC-SLI proposal):

[1] U. Wahl, J.G. Correia, E. Rita, J.P. Araújo, J.C. Soares, and the ISOLDE collaboration: "Fe and Cu in Si: lattice sites and trapping at implantation-related defects", Nucl. Instr. Meth. B 253 (2006) 167-171.

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6b) Publications related to IS453:

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