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

**Radiotracer diffusion in semiconductors and metallic
compounds using short-lived isotopes**

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Summary

The transport of atoms in solids is of central importance for solid state physics, chemistry, metallurgy, and material sciences. Since the mobility of atoms in solids contributes to many physical phenomena the study of diffusion processes is of fundamental interest for solid state physics. Diffusion processes were frequently investigated using radioactive isotopes (radio-tracers). The application of short-lived isotopes delivered at ISOLDE extends substantially the possibilities of investigating diffusion processes in solids. In particular, a new experimental set-up to be installed at ISOLDE in this year will enable the use of radioactive isotopes with half-lives down to minutes. Alternatively, in special cases diffusion processes can be investigated with help of hyperfine techniques on an atomic scale, like by perturbed $\gamma\gamma$ -angular correlation (PAC). Here, the motion of the atom of interest becomes visible directly via characteristic changes in the measured PAC spectra.

The experiments will contribute to the understanding of the following phenomena:

- Diffusion of dopants in compound semiconductors as a function of temperature and external partial pressures. Induced gradients of the deviation of stoichiometry cause internal electric fields. The influence of such internal electric fields on the diffusion of various types of dopants is of special interest.
- Self diffusion of Indium in Tri-indides structure using the PAC probe $^{117}\text{Cd}/^{117}\text{In}$.
- Self diffusion of Al atoms in Al and Al compounds. The use of ^{29}Al requires the new experimental set-up for on-line diffusion experiments.



1. Introduction

Diffusion processes are of essential importance for metallurgy and doping of semiconductors as well. The first application of radioactive isotopes for diffusion experiments dates back to 1920, when radioactive Pb atoms were used for ‘tracer diffusion’ by G. v. Hevesey to study self diffusion in lead [1]. Diffusion experiments can also be performed on atomic scale if the motion of the respective species of atoms is investigated directly. Such investigations are accessible with help of hyperfine techniques like perturbed γ - γ angular correlation (PAC). PAC investigations have been applied successfully for the study of interdiffusion in Triindides (see section 3.3). In semiconductors the understanding and the control of diffusion profiles of intrinsic and extrinsic defects is of central importance for developing electronic and optoelectronic devices with reduced structural sizes. At temperatures required for crystal growth and device processing, intrinsic point defects, like vacancies and self-interstitials, are highly mobile and can interact with extrinsic point defects, like dopant atoms. Common to most diffusion profiles in semiconductors reported so far is the monotonously decreasing concentration depth profile if the source of the diffusing species is located at the surface of the crystal and the diffusion length is small compared to the thickness of the crystal. However, in compound semiconductors large concentrations of intrinsic point defects can be achieved by inducing deviations from stoichiometry in the respective material. By exposing a compound semiconductor to the vapor pressures of one of its constituents, variations of the deviation from stoichiometry of the compound crystal as a function of depth and time can result in concentration gradients of intrinsic point defects, which can drastically influence the diffusion of defects, leading to anomalous diffusion profiles.

2. Experimental methods and previous work

An overview on the use of radioactive ion beams to explore solid state properties has been given by Forkel-Wirth [2]. An overview on the experimental techniques used for the identification of defects in semiconductors can be found in [3]. A review on the characterization of defects in semiconductors using radioactive isotopes can be found in [4].

Tracer diffusion

The principles of tracer diffusion studies have been unchanged since the first investigations by Hevesey [1]: First, a thin layer of material containing the tracer is deposited on the surface of the sample under study; alternatively, the tracer is implanted into the sample. Subsequently, the sample is heated for a predetermined time at a selected temperature. After cooling back to room temperature, the sample is sectioned into thin slices step by step and either the content of the radioactive tracer in each slice or in the remaining, unsectioned part of the sample is measured. From this measurement, the diffusion profile, i.e. the concentration of tracer atoms as a function of depth is determined. By repeating this experiment for different diffusion times and/or temperatures the diffusion parameters of the tracer, especially its activation enthalpy and pre-exponential factor are determined. From these values and the shape of the diffusion profile, conclusions about the diffusion mechanism can be drawn [5].

For radiotracer diffusion measurements, the availability and purity of radioactive ion beams at ISOLDE has enlarged the possibilities for studies of self- and impurity-atom diffusivities, considerably. For instance, the short-lived ^{31}Si isotope plays an important role in self-diffusion studies in group IV-IV compound semiconductors [6]. A similar case constitutes the

isotope ^{73}As in III-V compounds, where an unexpected interstitial diffusion mechanism has been revealed for the As self-diffusion in GaAs [7,8].

At the University of Saarbrücken, a high-vacuum chamber for tracer diffusion measurements has been constructed. This set-up will allow implantation, annealing, sectioning of the sample by ion-sputtering, and measurement of the radioactive tracers by detecting the decay-radiation, *in situ*. Changing the samples without breaking the vacuum will be possible, too. The set-up will allow the use of radioactive isotopes for tracer diffusion studies with half-lives down to minutes. After having tested the chamber at the University of Saarbrücken, the diffusion chamber will be installed permanently at CERN in this year.

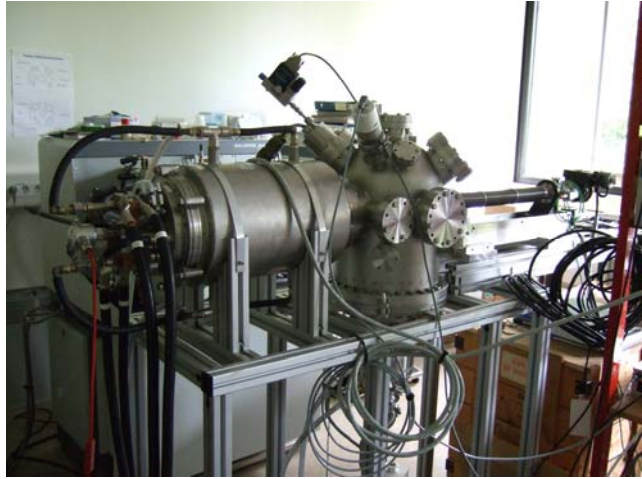


Figure 1: On-line diffusion chamber constructed at the university Saarbrücken. The chamber will be installed at ISOLDE within this year.

Perturbed $\gamma\gamma$ angular correlation (PAC)

An alternative approach to tracer diffusion is the measurement in real time of an observable that is sensitive to the discrete atomic jumps. One such observable is the electric field gradient (EFG) that can be detected by PAC. In general, microscopic insight into the structure and the thermodynamic properties of complexes (like formation probability, thermal stability) of complexes formed by interacting defects can be gained by detecting hyperfine interactions between nuclear moments of radioactive probe atoms and electromagnetic fields present at the site of the radioactive nucleus. These extra-nuclear fields arise due to the composition and geometrical arrangement of atoms of the immediate neighborhood of the probe atom. For nonmagnetic systems, two contributions to the hyperfine interaction have to be considered: the isomeric shift and the electromagnetic quadrupole interaction due to an electric field gradient (EFG). There exist several techniques for detecting such electromagnetic interactions using stable isotopes, like nuclear magnetic resonance (NMR) and electron nuclear double resonance (ENDOR). The use of radioactive probe atoms and techniques like the Mössbauer effect and the perturbed $\gamma\gamma$ or $e\gamma$ angular correlation spectroscopy (PAC) increases the experimental sensitivity by several orders of magnitude. Using the radioactive isotopes delivered by ISOLDE, these techniques, in particular PAC, have contributed to the characterization of defects and defect complexes in many semiconductors and metallic compounds (see [2,9] and references therein).

As mentioned above, an alternative approach to tracer diffusion the measurement in real time of an observable that is sensitive to the discrete atomic jumps. One such observable is the EFG detected at the nucleus of an atom on a sublattice having noncubic symmetry. If there is a reorientation of the EFG due to the jumps of neighbouring atoms, the spin rotation caused by the quadrupole interactions of the ensemble of tracer nuclei will lose coherence over time. With increasing sample temperature, diffusive motion first appears as damping or relaxation

of the static perturbation function in the PAC spectrum. The jump frequency w is defined as the inverse of the mean residence time of the tracer at a lattice site. Qualitative features of the relaxation process are described well by analytic approximations of the PAC perturbation functions as is given by Baudry and Boyer [10]. In their model the EFG has a constant magnitude and fluctuates among N different orientations and the EFG is zero averaging over all possible orientations. To obtain quantitative results for a wide range of jump frequencies w from PAC measurements (i.e. for a wide range of temperatures), the experimental PAC spectra are fitted using numerical calculations of exact functions by Winkler and Gerdau [11], improved by Evenson *et al.* [12]. These calculations cover different fluctuation regimes, i.e. different ratios between w and the EFG induced spin rotation frequencies.

3. Physics cases and proposed experiments

3.1 Diffusion in II-VI semiconductors

At temperatures required for semiconductor crystal growth and device processing, intrinsic point defects, like vacancies and self-interstitials, are highly mobile and can interact with extrinsic point defects, like dopant atoms and impurities. For this reason, detailed studies of the mobility and interaction of various defects have been performed in elemental semiconductors, like Si and Ge, and, but to a lesser extent, in various compound semiconductors [13,14]. Common to almost all diffusion profiles in semiconductors reported so far is the monotonously decreasing depth profile of the diffusers if the source of the diffusing species is located at the surface of the crystal. In contrast to Si and Ge, in compound semiconductors large concentrations of intrinsic point defects, as compared to thermally generated defects, can be obtained by inducing deviations from stoichiometry in the respective material. By exposing a compound semiconductor to the vapor pressures of one of its constituents, variations of the deviation from stoichiometry of the (e.g.) binary crystal as a function of depth and time are generated and, thereby, concentration gradients of intrinsic point defects.

Defects in semiconductors usually act as donors or acceptors present in different charge states. This property is used for forming *pn*-junctions, the most fundamental module for any electronic device. As a consequence, if an inhomogeneous distribution of charged defects is present, an internal electric field is generated acting on any charged particle present in the semiconductor. An internal electric field can also be generated exclusively by intrinsic defects if they act as donors and acceptors and are inhomogeneously distributed. Of particular technological importance is the fact that the diffusion of charged dopant atoms is strongly affected by the presence of an internal electric field [15].

The diffusion of Ag, and to lesser extent also of Cu, in CdTe was investigated during the last years, showing several very unusual properties depending on the sample conditions before diffusion and on the external conditions during diffusion [16,17,18].

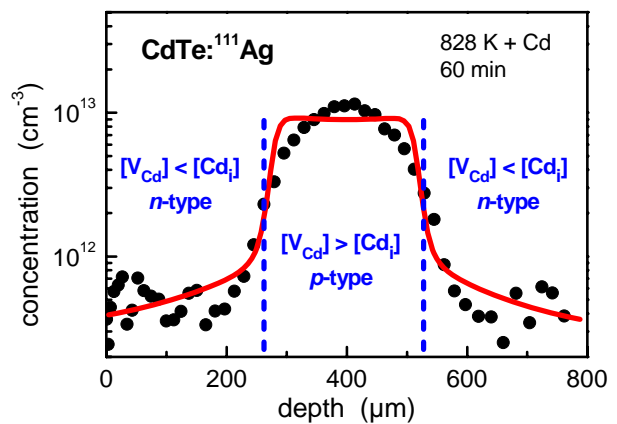


Figure 2: ^{111}Ag profile in CdTe measured after diffusion under Cd pressure. The red solid line is a simulation according to a theoretical model outlined in [19,20,21].

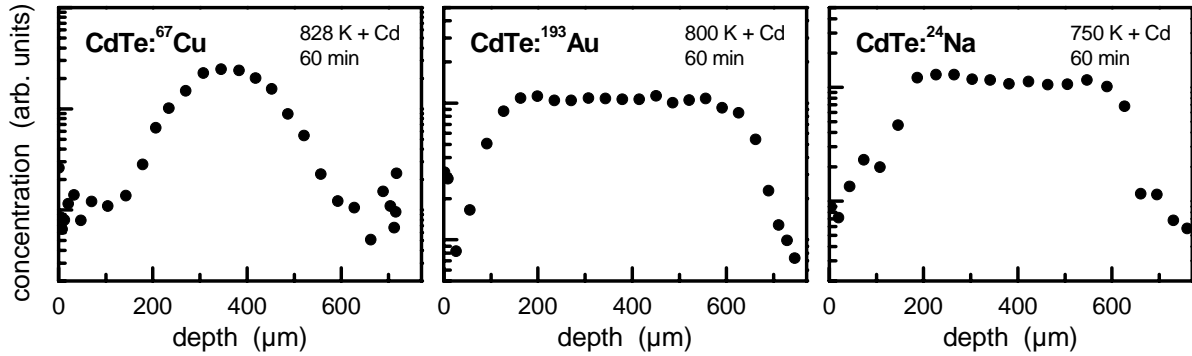


Figure 3: Concentration profiles for Cu, Au, and Na in CdTe after diffusion under Cd pressure.

The case of Ag diffusion in CdTe exhibits in a very pronounced way the effect of the internal electric field (generated essentially by the distribution of intrinsic defects) on dopant diffusion (Figure 2). Here, the important intrinsic defects are the interstitial Cd atom, which acts as a double donor, and the Cd vacancy, which acts as double acceptor. The distribution of these intrinsic defects can easily be manipulated by varying the external Cd partial pressure during diffusion. The CdTe crystal used for the experiment shown in Figure 2, initially, was Te rich, i.e. it contained a large concentration of acceptor like Cd vacancies, and was diffused under an external Cd pressure causing a large concentration of donor like Cd interstitials. The penetration depth of the Cd interstitials after the diffusion annealing extends about 250 μm from the surface into the crystal. In the limit of low dopant concentrations the shape of the profile is well understood from a theoretical point of view [19,20,21]. It turns out that the Ag atoms at diffusion temperature essentially are incorporated as highly mobile, positively charged interstitial donors. As a consequence, the Ag profile maps the electrical potential energy of charged particles in the CdTe sample.

By using the radioactive isotopes provided by ISOLDE, there have been identified more dopants (Cu, Au, Na) exhibiting essentially the same diffusion properties in $\text{Cd}_{1-x}\text{Zn}_x\text{Te}$ crystals (Figure 3). In addition, the diffusion of the transition metals Ni and Co seems to be strongly affected by the presence of an internal electric field, too, but the effect on the observed dopant profile differs strongly from the previous cases, like Ag, as is shown in Figure 4 [19,20]. The mechanisms leading to the profiles of Ni and Co, however, are not yet well understood theoretically.

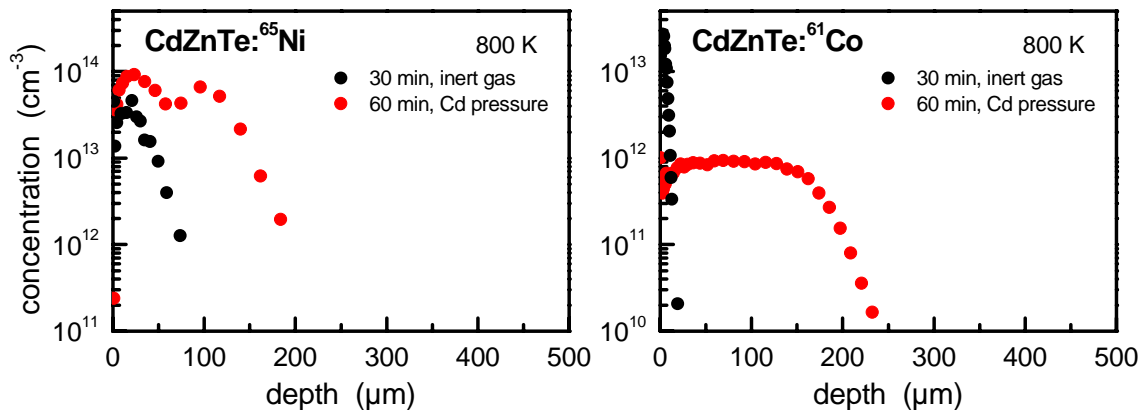


Figure 4: Concentration profiles for Ni and Co in CdZnTe after diffusion under inert gas atmosphere (black symbols) and under Cd pressure (red symbols).

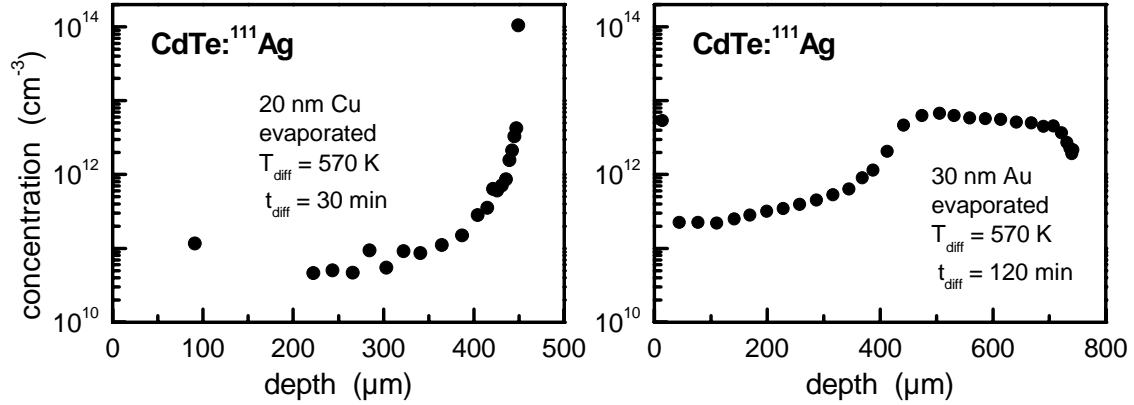


Figure 5: Ag diffusion profiles in CdTe obtained at the presence of an evaporated metal layer at $x=0$.

Besides the external Cd pressure the shape of the Ag profile in CdTe can strongly be affected by the surface properties of the crystal. These properties are modified in a very effective way by the evaporation of a metal layer onto the surface after the implantation process but before diffusion experiment. Figure 5 shows the Ag profiles in CdTe obtained after evaporation of a Cu (left) or Au (right) layer. It should be noted that the diffusion was performed at a quite low temperature of only about 570 K as compared to the previous cases. The effect of the metal layer appears to be more pronounced in case of Cu, but in both cases the Ag dopant is strongly pushed away from the evaporated layer. At present, the effect is assumed to be caused by the interaction of the atoms in the metal layer with those of the CdTe crystal. In this way, an effective source of Cd interstitials is generated by extracting Te atoms out of the CdTe crystal. It should be noted that an interaction depth of only a few monolayers is sufficient to explain qualitatively these experimental observations.

Diffusion data generally contain information about thermodynamic properties of the host crystal and of the dopant as well. In semiconductors, the effect of a possibly present internal electric field causes some complications regarding the quantitative understanding of the data, but on the basis of the present investigations it has been possible to develop a quantitative model enabling a careful analysis of unusual diffusion data as observed for Ag in CdTe. It should be noted that there is very few experimental information present in the literature regarding the influence of internal electric fields on diffusion processes. Investigating the dopant diffusion systematically as a function of temperature and external vapor pressure enables the determination of thermodynamic parameters mentioned above. In addition, the experimental data open up new possibilities for manipulating and designing dopant profiles by setting suitable external conditions during the diffusion process. Further, it is of general interest to find out to what extent diffusion processes in semiconductors are affected by the presence of internal electric fields, which are generated by the distribution of intrinsic and extrinsic defects. In the context of magnetic semiconductors, the possibility of shaping concentration profiles simply by a thermal diffusion process seems to be attractive, in particular.

Anomalous diffusion behavior might be expected to occur in other II-VI compound semiconductors, too, like CdS, ZnSe, and ZnO but also the III-V compound semiconductors. The effect seems to depend (i) on the possibility of generating an internal electric field of sufficient strength by appropriate gradients of intrinsic defects and (ii) on the existence of highly mobile charged impurities. In InP, SIMS measurements performed after simultaneous diffu-

sion of Zn and Cd show a drastically changed diffusion behavior as compared to the separate diffusion of Zn and Cd atoms. The data were interpreted by a substitutional-interstitial diffusion mechanism with a strong interaction between the diffusing species [22]. From our present understanding, however, this strongly modified diffusion behavior might be caused by the generation of internal electric fields.

We propose to continue and extend these diffusion studies in the II-VI semiconductors using the following radioactive dopants delivered by ISOLDE:

Isotope	$t_{1/2}$	Dopant nature in II-VI
^{24}Na	11.8 h	Group Ia acceptors
^{43}K	22.3 h	
^{67}Cu	61.9 h	Group Ib acceptors
^{111}Ag	7.5 d	
^{113}Ag	5.4 h	
$^{191}\text{Hg}/^{191}\text{Au}$	3.2 h	
$^{193}\text{Hg}/^{193}\text{Au}$	17.6 h	
^{67}Ga	3.2 d	Group IIIa donor
^{38}Cl	37 min	Group VIIa donors
^{77}Br	57 h	
^{71}As	65.3 h	amphoteric
^{69}Ge	39 h	deep state (high resistivity CdTe)
^{110}Sn	4.1 h	
^{65}Ni	2.5 h	Magnetic dopant
$^{61}\text{Mn}/^{61}\text{Co}$	1.6 h	
$^{103}\text{Cd}/^{103}\text{Pd}$	17 d	?

3.2 Self diffusion of Al atoms in Al and in Al compounds

Information about self diffusion in metals and metallic compounds are of fundamental interest for several kinds of process technology. Although Al is one of the most important materials for all days use, either as element or as constituent in various alloys, there is almost no information available about self diffusion of Al atoms. There are two reasons for the lack of information in this important point: Firstly, there exists only one stable isotope of Al, preventing diffusion experiments by techniques using stable isotopes, like SIMS. Secondly, there are no radioactive isotopes having half-lives, which would enable radiotracer diffusion experiments using conventional techniques for sectioning of the sample and subsequent measurement of radiation. The isotope ^{29}Al is the only one which comes into consideration for radiotracer experiments (the 10^6 y half life of ^{26}Al is not suited for radiotracer diffusion experiments because a number of about 10^{20} atoms would be needed), but the short half-life of only 6.6 min requires the development of a special technique for sectioning of the sample and measuring of the γ -radiation emitted from the sample.

At the University Saarbrücken a special setup for on-line diffusion experiments has been constructed (see section 4). Presently, this apparatus is under testing and will be installed at ISOLDE during this year. The design of the on-line diffusion chamber will enable the use of isotopes with half-lives down to several minutes for tracer diffusion experiments.

With this chamber, we propose to start investigations of self diffusion of Al in Al and Al compounds using the isotope ^{29}Al .

3.3 Indium diffusion in line compounds: Tri-indides with (A_3B) structure

Tri-indides formed with La, Ce, Pr, Nd, Gd, Er and Y have a L1_2 or Cu_3Au structure as shown in Figure 6. The crystal structure has a face centered cubic arrangement with La atoms at corners and In atoms at face centers of the conventional unit cell. The In site has tetragonal symmetry, so that the EFG is axially symmetric.

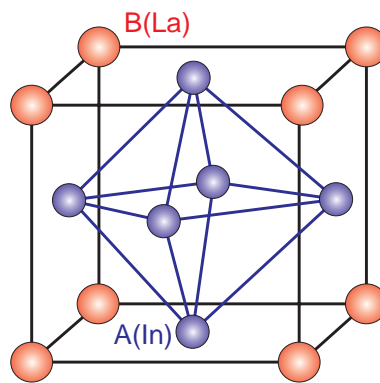


Figure 6: Crystal structure of In_3La , with In atoms at centers of cube faces.

These intermetallic compounds are “line compounds”, that is, intermediate phases that appear as vertical lines in binary phase diagrams [23] (see Figure 7).

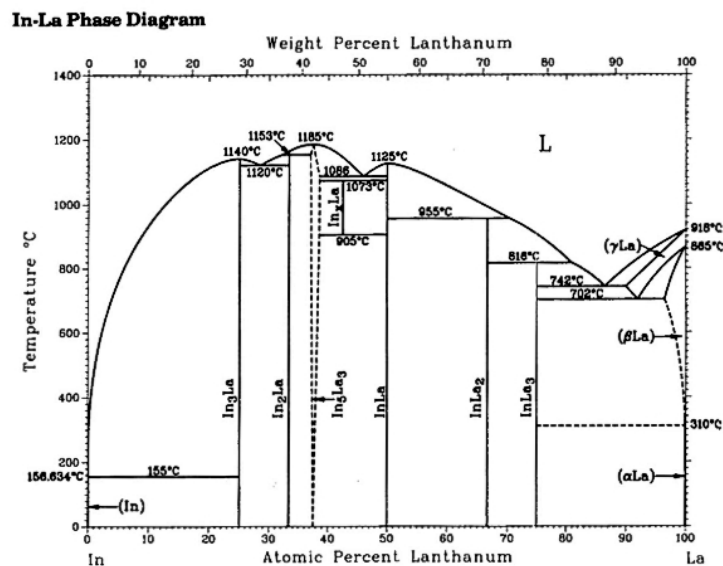


Figure 7: Phase diagram of the In-La intermetallic compound [23].

Although the line suggests a definite composition at some stoichiometric ratio of mole fractions of elements, intermediate phases must have fields of finite compositional extent at non-zero temperatures. Boundaries of a phase are determined by X-ray analysis as the com-

positions beyond which second phases begin to appear. The uncertainty in determinations of this type is typically 1 at%, so that line compounds generally may be assumed to have phase fields of indeterminate width less than or about 1 at.%. Usually, the field spans a stoichiometric composition, with deviations in composition on either side accommodated by the presence of structural point defects such as antisite atoms or lattice vacancies.

Using PAC with the probe atom $^{111}\text{In}/^{111}\text{Cd}$, we studied microscopically the atom movement of ^{111}Cd tracers on the In sublattice in In_3La [24] and in tri-indides formed with Ce, Pr, Nd, Gd, Er and Y [25]. For In_3La , the quadrupole interaction is axially symmetric with a frequency $\omega_Q = 11.34(1)$ Mrad/s at room temperature which describes the lattice EFG observed by ^{111}Cd on the In lattice site [26]. PAC spectra for the In-rich phase boundary composition (for which there are more La vacancies or In_{La} antisites) are shown in Figure 8.

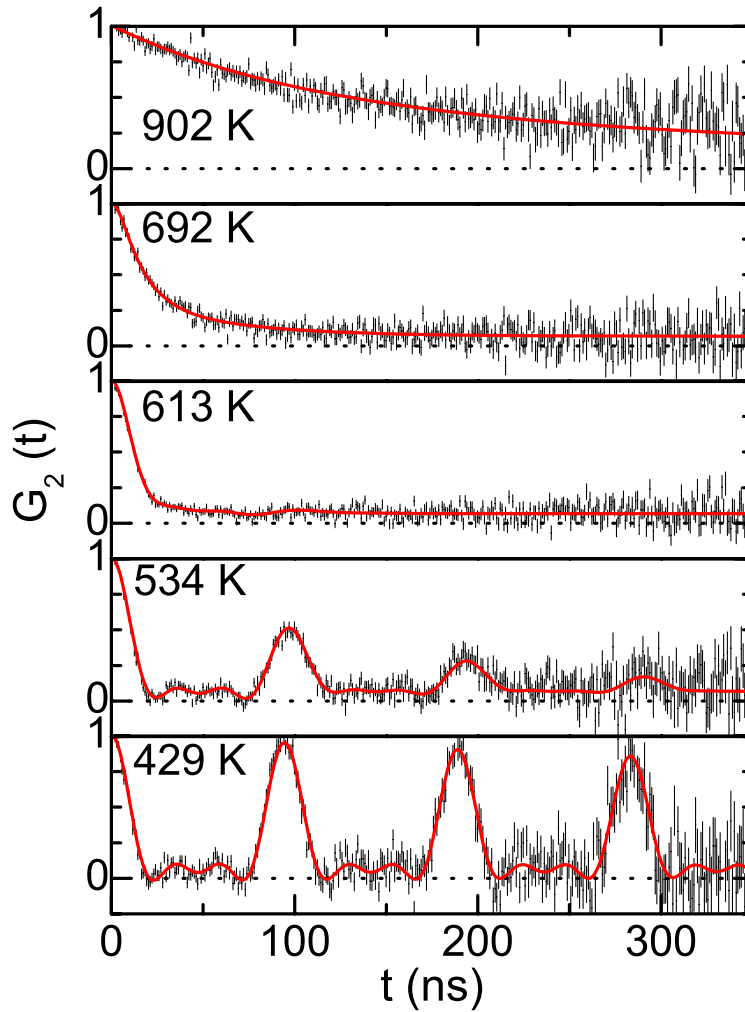


Figure 8: Perturbation functions $G_2(t)$ of ^{111}Cd tracer atoms in In_3La at different measurement temperatures [24].

At low temperature (429 K), the spectrum exhibits essentially the static quadrupole perturbation function. Significant relaxation is observed at 534 K that is attributed to jumping of Cd tracer atoms in the slow fluctuation regime. Maximum relaxation is observed at 613 K and above this temperature the perturbation function no longer exhibits the periodic quadrupole interaction frequency ω_Q . In the fast fluctuation regime above 613 K, the relaxation is ob-

served to decrease with increasing temperature due to motional averaging. From fits to the measured spectra the jump frequencies of the Cd tracer can be extracted [12].

While the phase In_3La appears as a line compound in the phase diagram, its phase field must have finite width, however small. Measurements on LaIn_3 samples purposely prepared to have the two, opposing phase boundary compositions were performed. Even though the compositions might differ by only about 0.1 at%, jump frequencies were observed to be 10-100 times greater at the more In-rich phase boundary than at the less In-rich boundary (Figure 9). Similar results were obtained for In_3Ce [25].

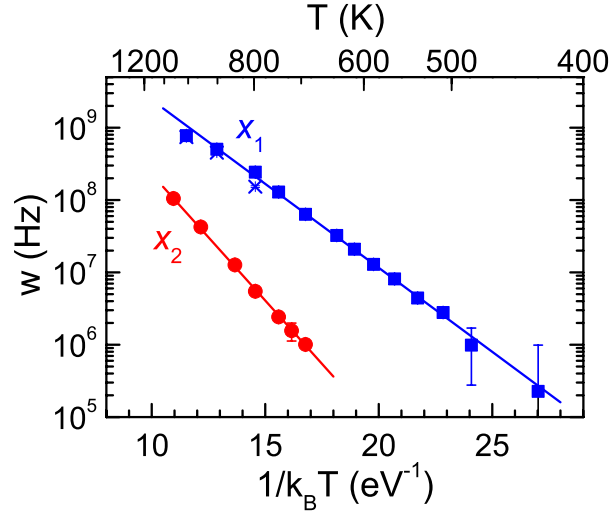


Figure 9: Temperature dependence of the frequency of reorientation of the EFG observed by the ^{111}Cd tracer atoms caused by jumps on the In sublattice in In_3La . The lines indicate fits of data for the two different phase boundary compositions x_1 (In-rich) and x_2 (In-poor) [24].

From the data shown in Figure 9, the migration enthalpy for the diffusion of Cd in In_3La and the diffusion mechanism involved can be identified. The jump frequencies w for the two compositions x_1 and x_2 were fitted with a thermally activated expression $w = w_0 \exp(-Q/k_B T)$ with $Q_1 = 0.53(1)$ eV and $Q_2 = 0.81(1)$ eV and the corresponding prefactors $w_{01} = 5.1(7) \times 10^{11}$ Hz and $w_{02} = 7(1) \times 10^{11}$ Hz [24].

All PAC measurements performed on these indides until now have used the probe atom $^{111}\text{In}/^{111}\text{Cd}$. That means, the observed diffusion on the In-sublattice is not diffusion of In but of Cd. An impurity Cd atom on the In-sublattice causes a local lattice distortion which might lead to an attractive or repulsive interaction with neighboring In-vacancies. This could influence thermodynamic properties such as the jump-frequency itself. We propose to use the PAC probe $^{117}\text{Cd}/^{117}\text{In}$ to perform jump-frequency measurements in tri-indides formed with La, Ce, Pr, Nd, Gd, Er and Y. With this probe, it is diffusion jumps of the daughter ^{117}In atoms that are observed. Such a measurement is analogous to a “self-diffusion” study in which the intrinsic behavior of the pure compound is measured.

4. Experimental requirements

At the University of Saarbrücken, a high-vacuum chamber for tracer diffusion measurements has been constructed. This set-up will allow implantation, annealing, sectioning of the sample by ion-sputtering, and measurement of the radioactive tracers by detecting the decay-radiation, *in situ*. Changing the samples without breaking the vacuum will be possible, too. The set-up will allow the use of radioactive isotopes for tracer diffusion studies with half-lives down to minutes. After having finished the tests at the University of Saarbrücken, the diffusion chamber will be installed at CERN, probably in April 2009. During the experiments at ISOLDE, the set-up requires a connection to an ISOLDE beam-line and space nearby for power supplies, control electronics and detectors.

The implantations of the radioactive isotopes needed for PAC measurements and tracer diffusion experiments using tracer with half-lives longer than about 1 hour will be performed in the solid state physics implantation chamber or the implantation chamber located on the high voltage platform. The sample treatment following the different implantations and the measurements will take place in the solid state physics lab at ISOLDE.

5. Beam time request

The great advantage of many of the beams required for this proposal is that they can be “shared” with other experiments. Typical collections for diffusion experiments in most cases need less than 1 hour after which the beam can be given to other users for gaps of often 1 shift or more. In addition to this, most of the collections here will be made in the solid state collection chamber on the GLM. This has the benefit that physics on the central beam line can proceed in parallel (as long as it’s a higher mass). In general, it would be appreciated if the accelerating voltage could be 60 kV. In recent years this has become something of a luxury at ISOLDE and the consequences for focusing the beam can be problematic for solid state physics. The desired number of atoms is typically between 10^9 and 10^{12} per sample depending on the half life. For half-lives greater than about 3 days, it may be desirable to ship samples back to the home institute in question.

We request a total of 18 isotopes over the next 2 years. Typically we would like to make several collections of each isotope which translates into approximately 1-2 shifts per isotope. In total therefore the total number of shifts requested is 25.

Isotope	$t_{1/2}$	Implantation energy
^{24}Na	11.8 h	60 keV
^{43}K	22.3 h	60 keV
^{67}Cu	61.9 h	60 keV
^{111}Ag	7.5 d	60 keV
^{113}Ag	5.4 h	60 keV
$^{191}\text{Hg}/\dots/^{191}\text{Pt}$	2.9 d	60 keV
$^{193}\text{Hg}/^{193}\text{Au}$	17.6 h	60 keV
^{67}Ga	3.2 d	60 keV
^{38}Cl	37 min	60 keV
^{77}Br	57 h	60 keV
^{71}As	65.3 h	60 keV
^{69}Ge	39 h	60 keV
^{110}Sn	4.1 h	60 keV
^{65}Ni	2.5 h	60 keV
$^{61}\text{Mn}/^{61}\text{Co}$	1.6 h	60 keV
^{29}Al	6.6 min	60 keV
$^{117}\text{Ag}/^{117}\text{Cd}$	2.5 h	60 keV
$^{103}\text{Cd}/\dots/^{103}\text{Pd}$	17 d	60 keV

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