

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

**Diffusion in Intermetallic Compounds
Studied Using Short-Lived Radioisotopes**

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Abstract

Diffusion – the long range movement of atoms – plays an important role in materials processing and in determining suitable applications for materials. Conventional radio-tracer methods for measuring diffusion can determine readily how distributions of radioactive probe atoms in samples evolve under varying experimental conditions. It is possible to obtain limited information about atomic jump rates and pathways from these measurements; however, it is desirable to make more direct observations of the atomic jumps by using experimental methods that are sensitive to atomic scale processes. One such method is time-differential perturbed γ - γ angular correlation spectroscopy (PAC).

Two series of PAC experiments using ^{111m}Cd are proposed to contribute to fundamental understanding of diffusion in intermetallic compounds. The goal of the first is to determine the dominant vacancy species in several $L1_2$ -structured compounds and see if the previously observed change in diffusion mechanism that occurs across the rare earth series in $R\text{In}_3$ (R = rare earth element) is accompanied by a change in dominant vacancy species. The goal of the second is to fully distinguish atomic jump rates on different sublattices in a material by measuring the inter- and intra-sublattice jump rates of Cd on the two Ga sublattices in Pd_3Ga_7 .

Requested shifts: 10 shifts, (split into 2 -3 runs over 2 years)



1 INTRODUCTION

Diffusion is of fundamental importance for processing metallic materials. This long range movement of atoms affects nucleation and growth, sintering, and homogenization in the formation of alloys and intermetallic compounds. Notable industrial applications directly influenced by diffusion are carburization and precipitation hardening. Other processing applications that depend on diffusion are welding, brazing, and surface modification treatments. After processing, consideration of diffusion properties is important for material selection.

In crystalline materials, a diffusing atom makes discrete steps among available positions in a lattice. Except for light elements such as H and Li, atoms usually move among regular lattice sites in intermetallic compounds, a process that requires empty spaces in the lattice, known as vacancy defects. In the usual potential-barrier model of atomic jumps, the jump rate w of an atom is given by $w = cw_0 \exp(-Q/k_B T)$ where w_0 is the attempt frequency, Q is the migration energy barrier, and c is a factor that describes the availability of neighboring sites to which the defect can jump. For diffusion of a tracer in a metallic system via a simple vacancy diffusion mechanism, the availability is given by $c = Z \cdot [V]$ where Z is the number of near neighbors on the tracer's sublattice and $[V]$ is the concentration of vacancies on that sublattice.

As a rule of thumb, energy barriers for jumps to first neighbor positions are much lower than those for more distant jumps, so that diffusion tends to be dominated by first neighbor jumps. An intermetallic compound has two or more sublattices. For a simply connected sublattice, *i.e.* for a sublattice with sites connected to other sites on the same sublattice by first neighbor jumps, diffusion of a tracer on that sublattice can progress via direct tracer-vacancy exchanges, which is referred to here as the simple vacancy (SV) mechanism. For a non-simply connected sublattice, the tracer must change sublattices, which results in the creation of temporary antisite defects, and is referred to here as a complex diffusion mechanism. An important example of a complex diffusion mechanism is the 6-jump cycle [1]. Determining the operative diffusion mechanism is one of the aims in diffusion studies of intermetallic compounds.

Conventionally, diffusion is studied by monitoring evolutions of concentration profiles of radioactive tracer atoms over a length scale of micrometers in single crystals, as described for example in Ref. 2. For lattices with cubic symmetry, the diffusion coefficient D is determined from such measurements, and the mean jump rate of the tracer atoms, w , can be determined from the relation $D = gfa^2w$, where g is a geometrical factor that depends on crystal structure and diffusion mechanism, f is a correlation factor that also depends on the diffusion mechanism, and a is the lattice parameter. In the case of self diffusion, *i.e.* when the radiotracer is the same as one of the elements of the host material, f is a constant; however when the tracer is an impurity, f is temperature-dependent and difficult to pin down experimentally.

Hyperfine methods, such as Mössbauer effect and nuclear magnetic resonance experiments, are sensitive to atomic jumps in the diffusion process and therefore provide an alternative means of studying diffusion [3,4]. Perturbed angular correlation of gamma-rays (PAC) is another hyperfine method that has been shown to be useful in the study of diffusion [5–13]. In PAC, signals in spectra become damped when the PAC probes switch lattice sites and when such jumps result in a change in the hyperfine interaction. The degrees of damping in PAC spectra are proportional to jump rates. Due to the limited number of available probe atoms, the hyperfine methods are unlikely to replace conventional diffusion measurement techniques. Nevertheless, there are situations for which the use of hyperfine methods is advantageous.

PAC has some noteworthy benefits. For example, it is not necessary to do PAC measurements in single crystals. Also, because the PAC measurement allows one to determine the lattice site(s) occupied by tracers, PAC measurements can be done in multi-component systems and can, in principle, distinguish jump rates on different sublattices. Moreover, PAC has the prospect of observing the vacancies and the other point defects that are involved in a diffusion process, which would provide a means of identifying diffusion mechanisms “directly”. In contrast to other hyperfine interactions techniques, the PAC spectroscopy itself does not depend on the measurement temperature, *i.e.* diffusion measurements can be performed up to the melting temperature of the material under study.

Two series of PAC experiments are proposed to contribute to fundamental understanding of diffusion in intermetallic compounds. One is to determine the dominant vacancy species in $L1_2$ -structured compounds to investigate whether or not the previously observed change in diffusion mechanism that occurs across the rare earth series in RIn_3 (R = rare earth element) [13] is accompanied by a change in the dominant vacancy species. The second is to demonstrate unequivocally that atomic jump rates among sites on different sublattices can be distinguished using PAC.

2 EXPERIMENTAL METHODS AND RELATED WORK

In PAC, the angular correlation between pairs of gamma rays emitted by an ensemble of nuclear probes is measured. When probes occupy a lattice site with non-cubic point symmetry, they experience an electric field gradient (EFG) and the angular correlation evolves in time due to the quadrupole interaction (QI) between the EFG and nuclear quadrupole moment of the probe. This time evolution appears as oscillations in the PAC spectrum. For additional information about the method, please see the book by Schatz and Weidinger [14] or review articles about the use of PAC at ISOLDE [15, 16].

The strength of the QI experienced by PAC probes often is denoted by the quadrupole interaction frequency ω_Q . When probes jump on a time-scale comparable to $1/\omega_Q$, the fluctuating QIs that accompany those jumps lead to damping and shifts in frequencies of the oscillating signal. Based largely on the work of Baudry and Boyer [17] and of Forker *et al.* [18], degrees of damping can be determined by fits of spectra to empirical fitting functions, and the degrees of damping in turn can be related to defect jump rates. Alternatively, the jump rates can be adjustable parameters in fit functions calculated using a stochastic model for fluctuating QIs, and the jump rates can then be determined in fits to spectra [19,20].

PAC has been used to study movement of the PAC tracer atom in a number of intermetallic compounds. Cd motion in β -Mn [6], Pd_3Ga_7 [9], Pt_3Ga_7 [10], $Al_{11}R_3$ ($R=La, Ce, Pr$) [11], $LaSn_3$ [7], RIn_3 ($R= La, Ce, Pr, Nd, Sm, Gd, Tb, Dy, Ho, Er, Tm, Lu, Sc, and Y$) [5, 8, 13], and RGa_3 ($R=Dy, Er, Lu$) [12] has been observed using the $^{111}In/^{111}Cd$ PAC probe. Notable findings from these works include a measurement of dissimilar degrees of motion-induced damping on two different sublattices in a material [9], that PAC can be used to identify when a complex diffusion mechanism is in operation instead of a SV mechanism by comparing jump rates at phase boundaries [5, 8], and that there is a change in diffusion mechanism for Cd across the rare earth series in RIn_3 [13]. An effort to study self diffusion in RIn_3 by using the $^{117}Ag/^{117}Cd/^{117}In$ probe using PAC is underway as a part of experiment IS489 [21].

3 PHYSICS CASES AND PROPOSED EXPERIMENTS

3.1 Diffusion mechanisms in $R\text{In}_3$ compounds (R =rare earth element and yttrium)

$R\text{In}_3$ compounds (R =rare earth element and yttrium) are cubic with the $L1_2$, or Cu_3Au , structure. The Au site has $m\bar{3}m$ point symmetry, which means that a PAC probe at that site will not experience an EFG ($\omega_Q = 0$). The Cu site has $4/m\bar{m}m$ symmetry with three possible orientations of the 4-fold rotation axis, as shown in figure 1. As a consequence, PAC probes at Cu sites experience an axially symmetric EFG and a probe jumping between adjacent Cu sites experiences a change in the orientation of the EFG. This means that a PAC experiment is sensitive to jumps of probes among the Cu sites. One expects a SV mechanism for tracers on the Cu sublattice because it is simply connected.

Extensive PAC measurements using the $^{111}\text{In}/^{111}\text{Cd}$ probe were made at varying temperature in $R\text{In}_3$ (R = rare earth, Sc, and Y) [5,8,13]. In all cases, no signals attributable to point defects such as vacancies were detected; only the axially symmetric signal of Cd occupying the In site was seen, and it was damped at elevated temperature due to motion of the Cd among In sites. Example spectra are shown in figure 2a for Cd in LaIn_3 at the In rich phase boundary. Fits to spectra using an appropriate stochastic model allowed the determination of QI fluctuation rates, some of which are shown in figure 2b.

As can be seen in figure 2b, the QI fluctuation rates and therefore the jump rates of Cd in In_3La are higher at the phase boundary with composi-

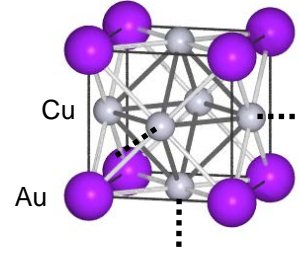


Figure 1. Conventional unit cell of Cu_3Au . The cubic unit cell is indicated by thin segments, and first neighbors are connected by wide segments. Dashed lines indicate the orientation of the 4-fold symmetry axes of Cu-sites.

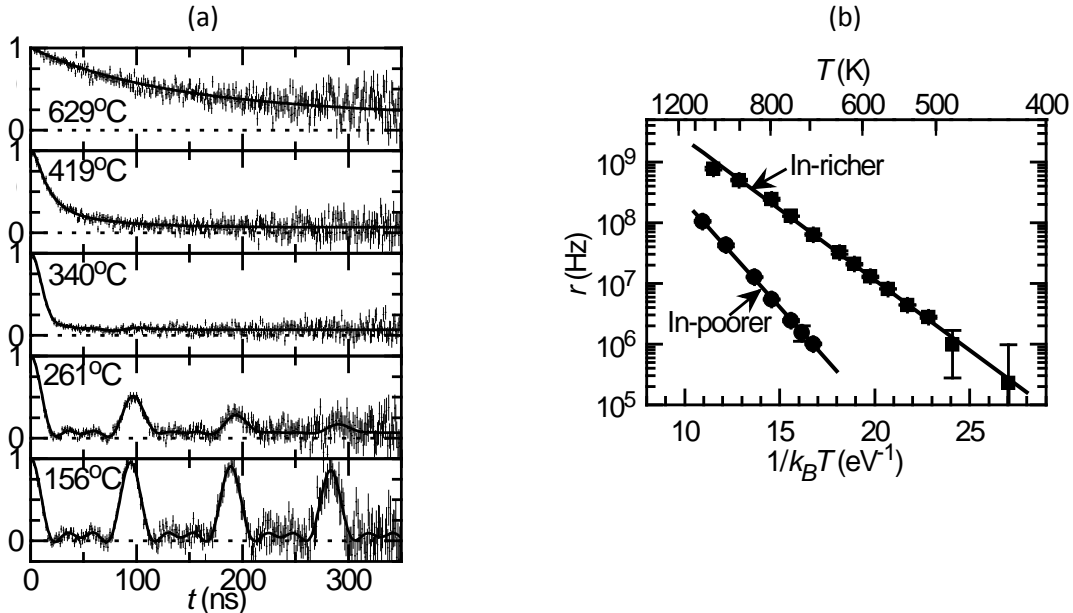


Figure 2. PAC spectra for $^{111}\text{In}/^{111}\text{Cd}$ in In_3La with composition at the In-rich phase boundary obtained at indicated measurement temperatures (a). QI fluctuation rates at the In richer and In poorer phase boundaries determined by fits to the XYZ stochastic model (b). Plots are of data from Ref. 5.

tion corresponding to an excess of indium than at the other phase boundary. If a SV diffusion mechanism is in operation, one expects that the jump rate should be lower at the In rich phase boundary. Therefore, a complex diffusion mechanism must be governing the diffusion of Cd in that system. For $R\text{In}_3$ with heavy rare earth elements (and with Y) on the other hand, the jump rates were found to be smaller at the In rich boundary indicating that the SV mechanism is in operation. A systematic study across the rare earth series showed a change in diffusion mechanism from a simple vacancy mechanism for the heavy rare earths to a complex diffusion mechanism for the light rare earths [13]. It was not possible to determine which type of complex diffusion mechanism is in operation or what the physical origin of the change in mechanism is. Below, a set of experiments are proposed to investigate the origin of this change in diffusion mechanism.

All the experiments described above were performed using the probe atom ^{111}In , which decays to the actual PAC probe ^{111}Cd . That means the interaction of the probe atoms with defects is determined by In, whereas the diffusion is observed after the decay of In to Cd. Using $^{111\text{m}}\text{Cd}$ as probe, both the interaction with defects and the diffusion are observed at the same probe atom.

Proposed experiment: $^{111\text{m}}\text{Cd}$ PAC in $R\text{In}_3$ ($R = \text{rare earth and Y}$). The goal of this set of experiments is to measure the QIs arising from intrinsic point defects attracted to the Cd probe in order to identify which point defects are present in largest concentrations. One possible explanation for the change in diffusion mechanism is that it is caused by a change in the dominant vacancy species, from an In vacancy for Y and the light rare earths to a rare earth vacancy for the heavy rare earths.

Because the parent of the PAC isotope in the earlier experiments using $^{111}\text{In}/^{111}\text{Cd}$ was a host element, the fraction of probes with at least one neighboring defect can be predicted using statistics of randomly distributed defects to be approximately $Z \cdot [\text{defect}]$ in the limit of small defect concentration where $[\text{defect}]$ is the defect concentration and Z is the number of near neighbors. Defect concentrations in the $R\text{In}_3$ compounds are thought to be less than 0.5% [5]. Therefore, the fraction of probes with nearby defects would be negligible, which is consistent with the lack of any signal attributable to defects in the earlier experiments.

In experiments using $^{111\text{m}}\text{Cd}$, the PAC probe is an impurity, in which case the fraction of probes with one nearby defect is given approximately by $Z [\text{defect}] \exp(E_B/k_B T)$ where k_B is Boltzmann's constant, T is the measurement temperature, and E_B is the probe-defect binding energy. For an attractive interaction, $E_B > 0$, and the fraction of probes with a neighboring defect is larger than what is found for randomly distributed defects. While it was not possible to observe defect signals in $R\text{In}_3$ using $^{111}\text{In}/^{111}\text{Cd}$, trapping of defects by $^{111\text{m}}\text{Cd}$ will lead to enhanced fractions of probes with neighboring defects.

In the proposed experiments, samples will be implanted with $^{111\text{m}}\text{Cd}$, annealed to remove implantation damage, cooled quickly to quench in an enhanced concentration of thermally activated defects, and measured using PAC at room temperature. Such a method involving an anneal and rapid cool to quench in excess thermally activated defects was used, for example, to produce complexes of In with Fe-vacancies in FeAl [22]. Once QIs involving defects have been measured, the identity of the defects will be determined by comparison of QIs predicted using density functional theory (DFT), in the same way that charge states of acceptors and Te vacancies near PAC probes in CdTe were determined [23].

3.2 Pd₃Ga₇

The Pd₃Ga₇ system is intriguing for the study of atomic jumps that occur among Ga sites. A portion of the crystal structure is shown in figure 3. There are two crystallographically inequivalent Ga sites, Ga(3) and Ga(4), so that the strength of an EFG measured at a Ga(3) site will be different than that measured at a Ga(4) site. The Ga(3) sites have $\bar{4}m2$ point symmetry, with three possible orientations of the 4-fold rotation axis in the (100) directions. The Ga(4) sites have $.3m$ point symmetry with rotation axes oriented in the four possible (111) directions. Therefore movement of PAC tracers from one Ga site to another can be detected because there is a corresponding change in EFG – either in orientation-only in the case of intra-sublattice jumps or in both orientation and magnitude for inter-sublattice jumps.

In previous ¹¹¹In/¹¹¹Cd PAC experiments [9], In was found to be distributed among Ga(3) and Ga(4) sites and not to occupy Pd sites. A selection of PAC spectra are shown in figure 4a. As can be seen, signals in the spectra are damped at elevated temperature. Spectra were analyzed using empirical fitting functions, and best-fit values of the damping parameters are shown in figure 4b. Activation energies of the signals corresponding to Ga(3) and to Ga(4) were found to be very similar, suggesting that diffusion on the two sublattices is coupled; however, it is difficult to understand why the damping factors differ by a factor of roughly 4.

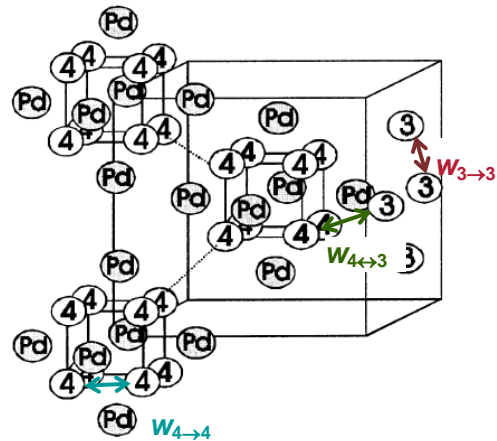


Figure 3. Partial conventional unit cell of Pd₃Ga₇. The Ga(3) sites are labeled simply by 3 and Ga(4) sites by 4. Representative jumps among Ga sites are indicated by double arrows and labeled according by jump rate.

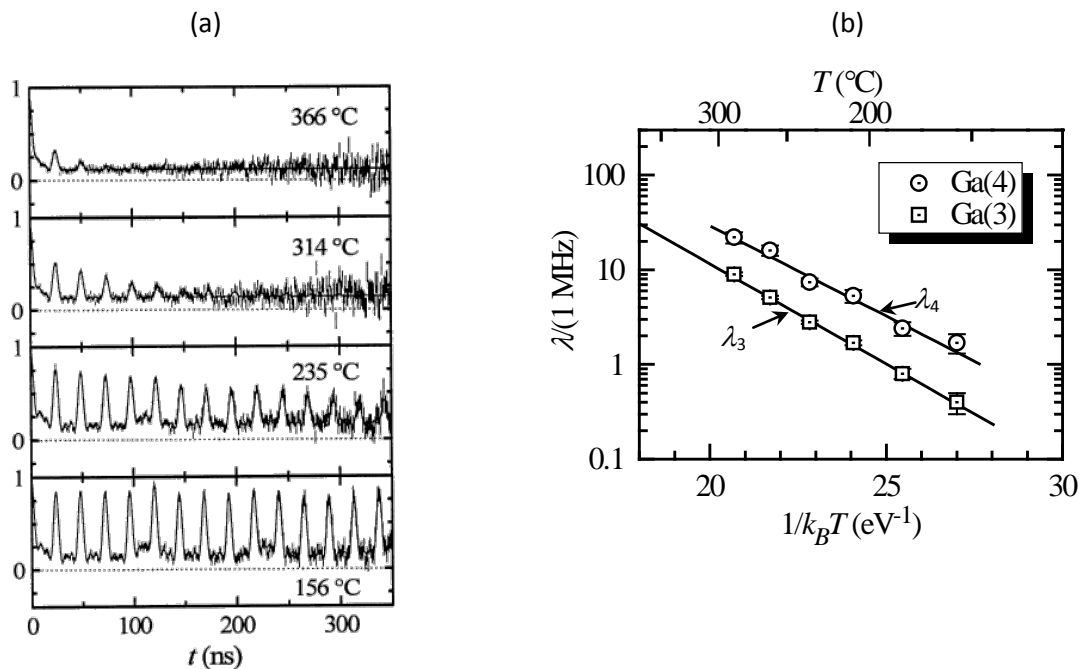


Figure 4. PAC spectra for ¹¹¹In/¹¹¹Cd in Pd₃Ga₇, obtained at indicated measurement temperatures (a). Fitted damping factors λ_3 and λ_4 for Cd occupying Ga(3) sites and Cd occupying Ga(4) sites, respectively (b). Plots are of data from Ref. 9.

In order to better understand the PAC data, a stochastic model for Cd jumping among Ga sites was developed and experimental spectra reanalyzed. The stochastic model had seven adjustable parameters:

- $f_{\text{Ga}(3)}$: fraction of probes in Ga(3) sites ($f_{\text{Ga}(4)}$ is not a free parameter because it is simply $1 - f_{\text{Ga}(3)}$)
- V_3 : magnitude of the EFG of a probe on the Ga(3) site with no nearby defects
- V_4 : magnitude of the EFG of a probe on the Ga(4) site with no nearby defects
- $r_{3 \rightarrow 3}$: rate at which an EFG changes from one particular orientation of principal axes corresponding to a Ga(3) site to one other orientation of a Ga(3) site
- $r_{4 \rightarrow 4}$: rate at which an EFG changes orientation from one Ga(4) site to another Ga(4) site
- $r_{3 \rightarrow 4}$: rate at which an EFG changes orientation from one Ga(3) site to one Ga(4) site
- $r_{4 \rightarrow 3}$: rate at which an EFG changes orientation from one Ga(4) site to one Ga(3) site

It was found that the stochastic model described individual spectra well; however, some parameters in the model were highly correlated, so that fitted values exhibited much scatter when plotted versus temperature – see for example, figure 5. An attempt to stabilize fit results by simultaneously fitting spectra obtained at different temperatures showed promise; however, some ambiguity remains in the interpretation of the results essentially because there are more model parameters than can be fitted reasonably to the experiment. A possible strategy to lift the ambiguity is to use results of defect total energy calculations using density functional theory. However, a more direct, independent experimental solution is desirable.

Proposed experiment: ^{111}mCd PAC in Pd_3Ga_7 . Performing PAC experiments using ^{111}mCd to measure Cd movement rather than the $^{111}\text{In}/^{111}\text{Cd}$ probe allows one to eliminate one of the adjustable parameters in the stochastic model. The fraction $f_{\text{Ga}(3)}$ corresponds to the distribution of the PAC parent (^{111}In in the previous experiments). Because of the principle of detailed balance, the fraction of the PAC daughter isotope on the Ga(3) site, $f^*_{\text{Ga}(3)}$, is given by $f^*_{\text{Ga}(3)} = 3r_{4 \rightarrow 3} / (4r_{3 \rightarrow 4} + 3r_{4 \rightarrow 3})$. When the PAC probe parent and daughter are the same chemically, as is the case for the ^{111}mCd probe, $f_{\text{Ga}(3)} = f^*_{\text{Ga}(3)}$, one of the reorientation rates can be eliminated from the fits. This should reduce the scatter in plots of reorientation rates such as the types shown in Fig. 5 and allow a precise determination of activation energies for each jump type.

In these experiments, samples will be implanted with ^{111}mCd and annealed to remove implantation damage. Each implanted sample will be used for one PAC measurement, and enough samples will be prepared to allow measurements at a selection of temperatures between 150 °C and 400 °C.

4 SAMPLE PREPARATION AND EXPERIMENTAL REQUIREMENTS

The ^{111}mCd will be implanted into samples in the solid state collection chamber on the GLM. The half-life of ^{111}mCd is only 48 min; therefore, post-implantation sample treat-

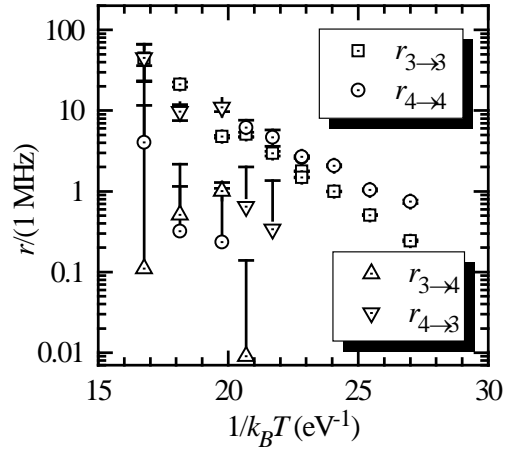


Figure 5. Fitted EFG reorientation rates for ^{111}Cd in Pd_3Ga_7 determined using a stochastic model of fluctuating EFGs.

ment and measurement will take place on-site in the solid state physics lab. All equipment needed for sample implantation, treatment, and measurement, are available already.

5 BEAM TIME REQUEST

We request 10 shifts of the ^{111m}Cd isotope in 2011 and 2012. The ^{111m}Cd will be implanted into samples in the solid state collection chamber on the GLM with about 10^{12} ions per sample. Other physics experiments on the central beam line can proceed in parallel (as long as they are utilizing isotopes with higher mass). The beam times can be shared with other experiments using the isotope ^{111m}Cd . If possible, it would be appreciated if an accelerating voltage of 60 kV could be provided to achieve a deeper implantation depth.

Summary of requested shifts:

Isotope	$T_{1/2}$	Target	Yield (ions/ μC)	Ion source	Shipping?
^{111m}Cd	48 min	Sn	2×10^8	HP (VADIS)	No

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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)*

Part of the Choose an item.	Availability	Design and manufacturing
SSP-GLM	<input checked="" type="checkbox"/> Existing	<input checked="" type="checkbox"/> To be used without any modification
Annealing furnaces in building 115	<input checked="" type="checkbox"/> Existing	<input checked="" type="checkbox"/> To be used without any modification <input type="checkbox"/> To be modified
	<input type="checkbox"/> New	<input type="checkbox"/> Standard equipment supplied by a manufacturer <input type="checkbox"/> CERN/collaboration responsible for the design and/or manufacturing
PAC setups in building 115	<input checked="" type="checkbox"/> Existing	<input checked="" type="checkbox"/> To be used without any modification <input type="checkbox"/> To be modified
	<input type="checkbox"/> New	<input type="checkbox"/> Standard equipment supplied by a manufacturer <input type="checkbox"/> 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			
	SSP-GLM	Annealing furnaces in building 115	PAC setups in building 115
Thermodynamic and fluidic			
Pressure	[volume][l]	N/A	N/A
Vacuum	<10 ⁻⁶ bar	N/A	N/A
Temperature	Room temp	600-1200 °C	150-400 °C
Heat transfer	N/A	N/A	N/A
Thermal properties of materials	N/A	N/A	N/A
Cryogenic fluid	[fluid], [pressure][Bar], [volume][l]	N/A	N/A
Electrical and electromagnetic			
Electricity	[voltage] [V], [current][A]	230V single phase	230V single phase
Static electricity		N/A	N/A
Magnetic field	[magnetic field] [T]	N/A	N/A
Batteries	<input type="checkbox"/>	N/A	N/A
Capacitors	<input type="checkbox"/>	N/A	N/A
Ionizing radiation			
Target material	Sn (HP –Vadis ion source)		
Beam particle type (e, p, ions, etc)	111m Cd ions		
Beam intensity			
Beam energy	60kV (desired)		

Cooling liquids	[liquid]		
Gases	[gas]	O ₂ , H ₂ , Ar	
Calibration sources:	^{111m} Cd		
• Open source	<input checked="" type="checkbox"/>		
• Sealed source	<input type="checkbox"/> [ISO standard]		
• Isotope	^{111m} Cd		
• Activity	~ 1MBq		Used for tuning detectors. Usually collected into Cd foil.
Use of activated material:			
• Description	Removal from chamber, transported to building 115 in standard Pb Castle shielding.	Annealed for 10 mins at temperatures up to 1200 °C in the furnaces in building 115	Placed in PAC machines in building 115 for measurement.
• Dose rate on contact and in 10 cm distance	No data in Swiss OPAP. To be discussed with CERN RP.		
• Isotope	^{111m} Cd		
• Activity	Max. 200MBq (per sample)		
Non-ionizing radiation			
Laser	N/A	N/A	N/A
UV light	N/A	N/A	N/A
Microwaves (300MHz-30 GHz)	N/A	N/A	N/A
Radiofrequency (1-300MHz)	N/A	N/A	N/A
Chemical			
Toxic	[chemical agent], [quantity]		
Harmful	[chemical agent], [quantity]		
CMR (carcinogens, mutagens and substances toxic to reproduction)	[chemical agent], [quantity]		
Corrosive		HCl, 100 ml	
Irritant	[chemical agent], [quantity]		
Flammable		Acetone, 250 ml	
Oxidizing	[chemical agent], [quantity]		
Explosiveness	[chemical agent], [quantity]		
Asphyxiant	[chemical agent], [quantity]		
Dangerous for the environment	[chemical agent], [quantity]		
Mechanical			
Physical impact or mechanical energy (moving parts)	N/A	N/A	N/A
Mechanical properties (Sharp, rough, slippery)	N/A	N/A	N/A
Vibration	N/A	N/A	N/A
Vehicles and Means of Transport	N/A	N/A	N/A
Noise			
Frequency	N/A	N/A	N/A
Intensity	N/A	N/A	N/A
Physical			
Confined spaces	N/A	N/A	N/A

High workplaces	N/A	N/A	N/A
Access to high workplaces	N/A	N/A	N/A
Obstructions in passageways	N/A	N/A	N/A
Manual handling	N/A	N/A	N/A
Poor ergonomics	N/A	N/A	N/A

0.1 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)

The power requirements are nothing out of the ordinary. All ovens and machines which are already installed in building 115 run off the standard single or three phase 230V line.