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Applications of radioactive ion beams to solid-state physics

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Abstract. Radioactive atoms have been used in solid-state physics and in material science for many decades. Besides their classical application as tracer for diffusion studies, nuclear techniques such as Mößbauer spectroscopy, perturbed angular correlation, β-NMR, and emission channeling have used nuclear properties (via hyperfine interactions or emitted α- or β-particles) to gain microscopical information on the structural and dynamical properties of solids. During the last decade, the availability of many different radioactive isotopes as a clean ion beam at ISOL facilities like ISOLDE/CERN has triggered a new era involving methods sensitive for the optical and electronic properties of solids, especially in the field of semiconductor physics. Extremely sensitive spectroscopic techniques like deep-level transient spectroscopy (DLTS), photoluminescence (PL), and Hall effect gain a new quality by using radioactive isotopes: Due to their decay the chemical origin of an observed electronic and optical behavior of a specific defect or dopant can be unambiguously identified. This review will briefly introduce the experimental techniques used and browse through the ongoing experiments in solid-state physics using radioactive ion beams demonstrating the wide variety of problems under study involving bulk properties, surfaces and interfaces in many different systems like semiconductors, superconductors, magnetic systems, metals and ceramics.

PACS. 61.72.-y Defects and impurities in crystals; microstructure – 71.55.-i Impurity and defect levels – 76.80.+y Mössbauer effect; other γ-ray spectroscopy

1 Introduction

Nuclear physics has developed a number of experimental techniques for detecting particles, γ-radiation and the interaction of nuclear moments with external electromagnetic fields that, meanwhile, have entered the field of condensed-matter physics with big success [1]. By employing radioactive nuclei, the ability of detecting signals from very small amounts of impurity atoms has turned out as an important advantage, particularly in characterizing the properties of semiconductors or surfaces. A further useful tool is represented by the nuclear transmutation process caused by the β-decay since this process effects a change of the chemical properties of the respective atoms in the same sample on a well-known time scale determined by the decay constant. This transmutation process represents an extremely useful analytical tool for the understanding of the opto-electronic properties of semiconductors. These materials possess a high sensitivity to the presence of small amounts of impurities and their chemical nature determines the electrical and optical properties.

Progress in semiconductor technology is driven by two requirements: developing new materials with unique optical or electrical features, and reducing the size of the individual constituents of an integrated device. These requirements demand a thorough understanding and control of defects in these materials; both of intrinsic defects, such as vacancies, self-interstitials, or anti-sites, and of extrinsic defects, such as dopants and impurity atoms. As a consequence, a strong effort is devoted to the investigation of defects and the electrical activation of dopant atoms. Experimental and theoretical techniques are needed for identifying the properties of defects, the diffusion mechanisms being responsible for the mobility of defects and the strengths of the mutual interactions between dopant atoms and intrinsic as well as extrinsic defects. In case of semiconductors, the electrical and optical properties are already significantly altered if 1 out of 10^9 atoms is replaced by a defect, which corresponds to a defect concentration of about 10^{14} cm^{-3}.

The routine analytical methods commonly applied to semiconductors are electrical and optical techniques, such as electrical transport measurements (Hall effect and conductivity), capacitance-voltage (C-V) measurements, deep-level transient spectroscopy (DLTS), and photolumi-

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nescence (PL). They are qualified by their high sensitivity to low defect concentrations, but they lack easily accessible microscopic information about the chemical identity of the respective extrinsic or intrinsic defects. The magnetic electron resonance techniques, EPR (electron paramagnetic resonance) and ENDOR (electron nuclear double resonance), along with their different varieties, represent an important exception if the defects constitute paramagnetic centers. In order to achieve the combination of high sensitivity to defects with the ability to identify the chemical identity of these defects, the employment of radioactive probes paves the way for new analytical techniques, such as the perturbed $\gamma\gamma$-angular correlation (PAC) technique, Mössbauer spectroscopy (MS), emission channeling (EC), and β-NMR. In addition, the sensitivity to low defect concentrations and at the same time to their chemical nature, is achieved by combining radioactive isotopes with chemically insensitive methods, such as Hall, DLTS, and PL.

Accelerator facilities, existing ones like ISOLDE (CERN, Geneva) and others that are on the brink of going into operation within the coming years, form the keystone for these analytical techniques, using radioactive probe atoms, since they provide a multitude of different isotopes. It is clear that the heavy investments for these facilities can only be justified if they are used by different scientific communities like particle physics, nuclear physics, atomic physics, solid-state physics, and life sciences. For ISOLDE, the distribution of the beam time available among the different research areas shows that the share of experiments in the area of solid-state physics meanwhile amounts to about 1/3. This article can only touch on the numerous results achieved by the different experimental techniques that require or can take advantage of radioactive ion beams in the field of condensed-matter physics. Recent overviews on the use of radioactive ion beams to explore solid-state properties are given by Wichert [2] and Forkel-Wirth [3]. An introduction to techniques using hyperfine interaction to characterize defects in semiconductors has been edited by Langouche [4]. An overview on the ongoing activities at ISOLDE is given in [5]. Finally, a very complete overview on most of the experimental techniques used for the identification of defects in semiconductors can be found in [6].

2 Nuclear probes in solids

2.1 Radioactive probe atoms and doping

Like stable isotopes, radioactive isotopes used as dopants influence the electronic and optical properties in materials like semiconductors according to their chemical nature. To take advantage of radioactive isotopes, their nuclear properties like half-lives, decay modes, decay energies, and nuclear moments have to be taken into account both for the solution of the problem under study and the experimental technique used. In principle, doping can be performed during the growth of the crystalline material, by diffusion, or by implantation. Only the last two procedures are normally used in case of radioactive dopants. The most versatile procedure is ion implantation: Having a variable implantation energy, the concentration of dopants, their lateral and their depth distribution can be controlled easily. Moreover, surface barriers, like oxide layers, or a reduced crystal quality of the near surface region play, in contrast to the diffusion procedure, no role and any unwanted co-doping by other elements is only determined by the purity of the ion beam and not by the normally much worse purity of the source material used for diffusion. Ion implantation is a process not limited by thermal equilibrium; therefore, doping is possible beyond any solubilities. Enumerating all these advantages, there have to exist some drawbacks: the energies used for implantation (keV to MeV) are much higher than typical binding energies of atoms in a crystal (eV) so that high concentrations of intrinsic defects (vacancies, interstitials, anti-sites, dislocations, even amorphous layers) are created. To achieve the desired properties, always a thermal annealing treatment of the implanted material is required in order to remove or at least to reduce the concentration of these intrinsic defects. At the required temperatures, several not intended processes can take place: unwanted diffusion of the implanted isotopes, decomposition of the material, and incorporation of unwanted impurities due to contamination of the equipment used for annealing. Radioactive isotopes with a half-life of at least one hour are normally required but some experiments can be performed with half-lives in the range of seconds. In this case, the only possible “sample treatment” is the variation of the implantation temperature. For practical reasons, like keeping the experimental conditions constant or restricting the concentration of the implants, the upper limit for usable half-lives is about one year.

2.2 Locating dopants

The electronic and optical properties of a semiconductor are not only determined by the chemical nature of a dopant but also by its location in the lattice. For decades, the most straightforward technique for locating impurity atoms within a lattice has been the so-called ion beam channeling effect [7], where an external ion beam (*e.g.*, a He beam with an energy of several MeV) is steered by small-angle Rutherford scattering along atomic rows or planes of the crystal (“channels”). Using this technique, the detection of impurities is limited to concentrations of at least 10^{18} cm^{-3}. The sensitivity of techniques based on the channeling effect can be drastically improved by using radioactive impurity atoms located in the crystal under study that emit charged particles (β^-, β^+, conversion electrons, α-particles), the so-called emission channeling (EC) technique [8–10]. Detecting the emission yield of these particles along different major lattice directions results in different emission yields compared to the observation along a random lattice direction. For the case of electrons, an enhanced emission yield along a certain lattice direction (“channeling”) is always the result of an emitting atom residing on or near this lattice row which guides the electrons toward the surface. A reduced yield or the absence of an increased yield along a major axis ("blocking") hints at an interstitial site of the emitting atom. The reverse is true for the case of positively charged emitted particles. During the last fifteen years, the EC technique has been applied to locate many different dopant atoms in Si, Ge and diamond, III-V semiconductors like AlN, GaN, GaP, GaAs, GaSb, InP, InAs and InSb, and II-VI semiconductors like ZnSe, ZnTe and CdTe (see [8–11] and references therein).

Along with the elements Fe, Ni, and Co, the element Cu is the most common unwanted transition metal impurity playing a role in manufacturing Si devices for it interacts with various dopants and other defects. Positively charged Cu is the fastest known interstitial diffuser in Si. At high temperatures the solubility of Cu in Si is the highest among all transition metals but it is negligible at room temperature. As a consequence, Cu shows a strong tendency to react with various defects and to form precipitates. The development of new position-sensitive detectors for electrons in the 40–1000 keV energy range [12] increased the detection efficiency of electron emission channeling roughly four orders of magnitude compared to ion beam studies, and about two orders of magnitude compared to EC using conventional detection. Together with the development of laser ionization sources for transition metal isotopes at ISOLDE [13], the first EC experiments on the lattice location of Cu in Si have recently become possible [14]. Using the same experimental setup, Wahl *et al.* [15] studied the lattice site of Er in Si. Er forms luminescent centers in Si and may allow the production of Si-based optoelectronic devices. Using the isotope ^{167}Tm decaying to $^{167\mathrm{m}}$Er, the authors proved that Er is located on near-tetrahedral interstitial lattice sites in Si.

2.3 Neighborhood of dopants

To act as electrically active donor or acceptor, the lattice around the dopant atom has to be as perfect as possible. The presence of lattice defects like vacancies can suppress the doping effect. Donor or acceptor atoms form positively and negatively charged ions, respectively, in the lattice. This charge leads to a Coulombic attraction towards the oppositely charged lattice defects or impurity atoms present in the material. If such defects start to diffuse, *i.e.* during the annealing procedure after ion implantation, they can be catched ("trapped") by the dopants resulting in the formation of dopant-defect pairs —so-called donor-acceptor pairs (DAP)— or more complicated complexes consisting of more than two constituents. This process results in the reduction of the carrier concentration present in the semiconductor ("passivation"). A microscopic insight into the structure and the thermodynamic properties (formation probability, thermal stability) of such complexes can be gained by detecting the hyperfine interaction between the nuclear moments of radioactive dopants and the electromagnetic fields present at the site of the radioactive nucleus. These fields are caused by the composition and the structural arrangement of the immediate neighborhood of the dopant. For the case of nonmagnetic semiconductors, two contributions to the hyperfine interaction have to be considered: the isomeric shift (IS) and the electromagnetic quadrupole interaction due to an electric field gradient (EFG). There exist several techniques for detecting this latter hyperfine interaction using stable isotopes, like nuclear magnetic resonance (NMR) and electron nuclear double resonance (ENDOR). But once more, the use of radioactive probe atoms increases the experimental sensitivity by several orders of magnitude. Especially the NMR technique can detect defect concentrations as low as 10^8 cm^{-3} by using polarized radioactive ion beams implanted into the semiconductor and by detecting the hyperfine interaction via the β-asymmetry of the decay radiation of the implanted radioactive probe atom [16].

The basic idea of the Mössbauer effect is the recoil-free emission of γ quanta from radioactive nuclei embedded in a solid. If the emitted γ quantum populates the ground state of the nucleus, it is possible to resonantly absorb a recoil-free emitted γ quantum (source) by the same nucleus in its ground state (absorber). To get a reasonable sensitivity, the system under study acts as the host matrix of the source atoms and is compared to a standard absorber. The electric charge distribution (electrons and nucleons) around the probe nucleus creates a shift of the resonance energy due to the electric monopole interaction and gives rise to the isomer shift (IS), which is only accessible by the Mössbauer effect. The information to be extracted from the isomer shift is the lattice site of the Mössbauer atom and its charge state. The latter parameter indicates, for example, whether the probe atom in a semiconductor acts as a donor and has emitted its electron into the conduction band. An overview of the ongoing work in semiconductors and metals using ME together with the radioactive ion beams delivered by ISOLDE can be found in [17].

If the charge distributions around a nucleus in a lattice has a symmetry lower than cubic, an electric field gradient (EFG) is created at the site of the nucleus. This situation exists in noncubic crystal lattices and at the presence of defects in the neighborhood of the probe atom. The EFG, defined as the second spatial derivative of the electric potential and, therefore, being a tensor, contains information on the symmetry and the orientation of the charge distribution with respect to the crystal axis, and delivers information on the configuration of the defect causing the EFG. It interacts via the hyperfine interaction with the nuclear quadrupole moment of the probe nucleus and leads to a quadrupole splitting ΔE of the m-substates of the involved nuclear levels (electric quadrupole interaction). A technique being especially suited for detecting the EFG is the perturbed $\gamma\gamma$- or e$^-\gamma$-angular correlation (PAC) spectroscopy. Here, the frequency $\omega = \Delta E/\hbar$ of the precession of the nuclear spin $\mathbf{I}$ is measured, which depends on the magnitude of the EFG. In addition to the EFG, the spin $\mathbf{I}$ and the nuclear quadrupole moment Q of the intermediate state of the daughter isotope define the value of the measured frequency ω.

The existence of intrinsic (vacancies or self-interstials) and extrinsic (impurity atoms) defects can be inferred from the complexing of defects with probe atoms, because the different types of defects give rise to a unique IS or EFG at the site of the probe atom. In contrast to metals, in semiconductors the formation of pairs is strongly enhanced if donors and acceptors are involved. In this case, the mutual Coulombic attraction supports the formation of donor-acceptor pairs (DAP) considerably. Due to the high sensitivity of the hyperfine interaction techniques to defects, it has been possible to clearly discriminate the presence of extrinsic and intrinsic defects, which is often a fundamental problem in defect studies. There is a large number of reports on intrinsic and extrinsic defects, trapped at radioactive probe atoms and detected by the PAC [18]. Extrinsic defects comprise impurity atoms that are either dopants, which are intentionally introduced, or contaminants, which are unintentionally introduced during different processing steps and during the growth of the respective crystal. It should be noted that there are recent *ab initio* calculations, which are able to account with high accuracy for the EFG caused by the formation of DAP on substitutional lattice sites in Si, Ge [19] and in CdTe [20]. This new development opens up the possibility of a unique assignment of defects to measured EFG values and thereby to a unique identification of defects.

Especially, interstitial impurities such as transition metals and hydrogen, most easily influence the properties of semiconductors due to their high mobility. Among the interstitial impurities, playing a role in context with the electronic and optical behavior of semiconductors, hydrogen represents a very important impurity in all semiconductors, both from the technical and the scientific point of view. During various manufacturing steps hydrogen is easily unintentionally incorporated into semiconducting material where it very efficiently interacts with extrinsic and intrinsic defects. During the last ten years, PAC experiments have been performed to study H in Si [21] and III-V semiconductors [22]. A considerable amount of new information could be provided concerning formation, microscopic structure and stability of acceptor hydrogen complexes. In addition, PAC experiments on free hydrogen diffusion in III-V semiconductors, for the first time, opened a microscopic insight into this process by directly observing the diffusing H atom [23].

Another example for the sensitivity of the PAC technique is the study of the doping of the high-T_c superconductor $YBa_2Cu_3O_{6+\delta}$ (YBCO) with Hg which may change the superconducting properties of the material. In this case, the isotope ^{197m}Hg produced by ISOLDE was implanted into YBCO and used for $e^-\gamma$ PAC measurements. The results [24] showed that the Hg atoms occupy unique lattice sites. From the possible lattice sites, the Ba/Y sites could be excluded.

2.4 Electrical and optical properties

From the application point of view, the electrical and optical properties of a semiconductor as determined by the presence of dopants and defects, decide on the feasibility of a device. To determine these properties, a set of very accurate techniques has been developed: Hall effect measurements, photoluminescence spectroscopy (PL), capacitance-voltage measurements (C-V), and deep-level transient spectroscopy (DLTS) [25]. All of them are perfectly feasible without any radioactive isotope, but they have severe problems to identify the chemical nature of the involved defects.

The most obvious quantity characterizing a semiconductor is its electrical conductivity which is determined by the type of majority charge carriers present, their concentration, and their mobility. These quantities can be determined by measuring the specific conductivity and the Hall effect as a function of temperature. The theoretical basis and the experimental realization is described in all introductory books on solid-state physics; a more thorough discussion can be found in [26,27].

Photoluminescence spectroscopy (PL) is a standard optical characterization technique in semiconductor physics [28,29]. At low temperatures, laser light with an energy exceeding the energy of the band gap is used to excite electrons from the valence band into the conduction band. These electrons relax to the band edge of the conduction band diffusing within the sample, and finally search their way back to the valence band. The corresponding optical transitions can be a band-band transitions *(e, h)*, but can also involve states within the band gap, like a transition between a donor and acceptor state *(D, A)* and a transition from the conduction band into an acceptor state *(e, A)*. If the transitions are not saturated, the intensity of the detected PL light corresponds to the concentration of the observed defects.

Despite their ability to characterize the properties of dopants and defects in semiconductors, one has to recall that these techniques are not able to directly determine the chemical nature of a defect. Hence, the assignments found in the literature are sometimes controversial. By using radioactive isotopes, one way out of this dilemma is to use element specific properties, like the half-life of a radioactive isotope undergoing a chemical transmutation. If a change in conductivity or an optical transition are due to a defect in which the parent or daughter isotope is involved, the concentration of that defect will change according to the half-life of the radioactive decay. This time-dependent change of the defect concentration will show up in the corresponding spectroscopic signal adding the lacking chemical information to the data delivered by these standard semiconductor characterization techniques. During the last decade, radioactive isotopes have been used in combination with DLTS [30,31], Hall effect [32,33], C-V [34], and PL [35–37] measurements to characterize the optoelectronic properties of many dopants in elementary and compound semiconductors.

2.5 Tracer diffusion

The development of new materials for technical applications (metals, alloys, semiconductors, ceramics) is strongly correlated with the progress in understanding the diffusion mechanisms of atoms in solids [38]. Without an intimate experimental knowledge of the diffusional behavior of impurity atoms accompanied by the understanding of the underlying atomic mechanisms, the manufacturing of modern, highly integrated electronic circuits with structural dimensions down to 100 nm would be impossible. The principles of tracer diffusion studies have been unchanged since the first investigation of the diffusion of ^{209}Pb in $PbCl_2$ by Hevesy [39] in 1920: First, a thin layer of material containing the tracer is deposited on the surface of the sample under study. Subsequently, the sample is heated for a fixed time at a certain 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 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 several 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 [40]. Nowadays, using Ar ion sputtering, sectioning of monolayers is possible yielding a much better depth resolution. In this way the possibility exists for diffusion studies either at low temperatures or with very short annealing times at elevated temperatures enabling the use of radioactive isotopes with half-lives of minutes. The use of ion beams for introducing the radioactive tracers just below the surface of the sample (about 10–50 nm for 60 keV ion energy) has several advantages [41]: diffusion barriers at the surface like oxide layers can be avoided and the mass-separated beam is isotopically very clean, allowing experiments at very low impurity concentrations.

A recent example is given for semiconducting alloys formed by the mixture of Si and Ge. By changing the mixing ratio, the size of the band gap can be tuned continuously between 0.66 eV (pure Ge) and 1.12 eV (pure Si) and in contrast to other compound semiconductors with tunable band gap like $Al_xGa_{1-x}As$, the existing silicon technology can be used for building the new devices. In spite of the great similarity of the group-IV semiconductors Si and Ge, their intrinsic diffusion behavior as controlled by point defects is very different [42]. In Ge, vacancies are the defects that control the self-diffusion at all temperatures, whereas in Si vacancies control the diffusion only at temperatures below 1000 °C. At higher temperature the self-diffusion is driven by the presence of Si interstitial atoms. An especially peculiar diffusion behavior is demonstrated by Au both in Si and Ge. Almost all Au atoms reside on substitutional lattice sites whereas only a very minor fraction occupies interstitial lattice sites. But this latter fraction can diffuse very rapidly and is dominating the diffusion process. At the same time, there has also to exist a continuous exchange of Au atoms between substitutional and interstitial sites. This exchange can be either controlled by Si self-interstitials or via vacancies. These two mechanisms lead to different shapes of the diffusion profile. Using the tracer ^{195}Au implanted at ISOLDE, Fischer *et al.* [43] studied the diffusion of Au in relaxed $Si_{1-y}Ge_y$ layers. The effective diffusion coefficient depends on the composition of the SiGe. Above a Ge content of 9%, the diffusion coefficient measured at 900 °C is drastically reduced. It was also shown that for Ge concentrations below 10%, the Au diffusivity is controlled by the self-intersitial component present in the alloy whereas between 10% and 24% Ge content the diffusivity of the interstitial Au component dominates.

3 Nuclear probes on surfaces and in interfaces

Using the PAC technique, information on the local structure, *e.g.* the site occupied by a probe atom on the surface, can be obtained via the EFG. For magnetic systems, the PAC can be used to detect its magnetic properties via the magnetic hyperfine interaction between the magnetic field at the site of the probe atom and its nuclear dipole moment. PAC measurements require only 10^{-4}-10^{-3} of a monolayer of radioactive probe atoms, therefore the macroscopic properties of the layer containing the probe atoms are not influenced by the presence of the probe atoms. In ultrathin magnetic multilayered structures, the magnetic properties can vary from layer to layer. Magnetic properties of surfaces and interfaces have been studied using a dedicated UHV chamber (ASPIC) allowing the use of the radioactive ion beams delivered by ISOLDE [44]. Aside from containing the usual equipment for surface characterization like LEED and AES, ASPIC allows that the radioactive probe atoms can be catched and deposited on surfaces with thermal energies. In addition, atomic layers of two different materials can be grown on the surface of a crystal by molecular beam epitaxy. This allows the study of multilayer systems. An overwiew of the measurements performed at ISOLDE is given in [44].

Some nonmagnetic elements get magnetically polarized in contact with a ferromagnetic material. This observation triggered experiments in multilayer systems with alternating layers of magnetic and nonmagnetic material. Using ASPIC, experiments on the system of ferromagnetic Ni in contact with paramagnetic Pd have been performed using the probe ^{111}Cd [45,46]. If the impurity is incorporated in the topmost Ni layer, the magnetic hyperfine field is reduced by 50% compared to the bulk value. If the probe atoms are incorporated in a Pd layer grown on top the Ni(001) crystal, static magnetic hyperfine fields have been observed clearly proving that a magnetic order is induced in Pd by the ferromagnetic Ni substrate. Positioning the probe atoms in the second Pd monolayer, the magnetic hyperfine field is considerably reduced.

An even more direct use of radioctive probe atoms absorbed on a surface has been demonstrated by Ashkenazy *et al.* [47]. They used the neutrino recoil generated during the electron capture decay of ^{107}Cd to determine the Cd site on a structured Ni surface.

4 Conclusion

The techniques and examples discussed in the context of defects in solids and magnetic properties of surfaces and interfaces demonstrate that radioactive isotopes play an essential role in the field of nuclear solid-state physics. Mostly produced by particle accelerators, they provide as nuclear probes unique information about their local surroundings on an atomic scale. In particular in semiconductors pyhsics, they constitute an ideal tool for the determination of chemical nature, lattice location, thermodynamical properties, dynamical, electronic and optical behavior of intrinsic and extrinsic defects with very high sensitivity.

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