Radioactive ion beams and techniques for solid state research

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Abstract

In this paper we review the most recent and new applications of solid state characterization techniques using radioactive ion beams. For such type of research, high yields of chemically clean ion beams of radioactive isotopes are needed which are provided by the on-line coupling of high resolution isotope separators to particle accelerators, such as the Isotope Separator On-line (ISOLDE) facility at CERN. These new experiments are performed by an increasing number of solid state groups. They combine nuclear spectroscopic techniques such as Mössbauer, Perturbed Angular Correlations (PAC) and Emission Channeling with the traditional non-radioactive techniques like Deep Level Transient Spectroscopy (DLTS) and Hall Effect measurements. Recently isotopes of elements, not available before, were successfully used in new PAC experiments, and the first Photoluminescence (PL) measurements, where the element transmutation plays the essential role on the PL peak identification, have been performed. The scope of applications of radioactive ion beams for research in solid state physics will be enlarged in the near future, with the installation at ISOLDE of a post-accelerator device providing radioactive beams with energies ranging from a few keV up to a few MeV. © 1998 Elsevier Science B.V.

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1. Introduction

During the year 1996, the Centenary of the Discovery of Radioactivity was commemorated around the world and motivated the reflection about the role of nuclear physics in fundamental and applied research. One year later it is still appropriate to discuss in particular, the usefulness

of new applications of nuclear techniques to solid state physics, which rely nowadays on the on-line production of a wide variety of radioactive ion beams.

The first applications of radioactive isotopes to solid state physics started a long time ago in the late 1920s, with the first radio tracer diffusion experiment [1]. In the late 1940s at the same time that the first “industrial” mass separator for radioactive ion beams was developed for the Manhattan project [2], researchers at Bell Labs proposed the use of ion doping techniques to process silicon based transistors [3].

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The use of nuclear reactors has proved to be an easy way to produce radioactive isotopes by neutron irradiation and activation of targets used for off-line implantation, or by in-situ activation of impurities which were previously introduced into the materials. Both methods are still routinely used today on materials hardness and diffusion studies [4]. However such studies are limited both by the neutron capture cross-sections and the small number of long-lived isotopes needed in off-line implantation experiments.

More sophisticated techniques that rely on specific properties of excited nuclei were introduced in solid state research in the late 1960s. The so-called “Hyperfine Interactions” (HI) techniques, which have been developed to investigate the nuclear structure phenomena, are used today to probe the local fields within the materials via its interactions with the nuclear moments. Mössbauer Effect (ME) [5], Perturbed Angular Correlations (PAC) [6], Nuclear Orientation (NO) [7] and the β -Nuclear Magnetic Resonance (β -NMR) [8] are examples of such techniques. The applications, however, are restricted by the limited number of adequate probe isotopes, particularly for off-line experiments. In spite of that, a small number of appropriate nuclei, with half-lives of the order of several hours, stimulated new experiments oriented for studies of impurity doping on bulk, surfaces and interfaces in metals and semiconductors [9–11].

Nowadays, the success of nuclear solid state physics research depends on the availability of a broad range of appropriate radioactive isotopes, on an adequate sample preparation, and of a good interaction between the solid state community and the physicists who have the know-how of the nuclear techniques. The on-line production of radioactive isotopes is the technical approach that best fulfills the first of these requirements. Several such radioactive facilities exist or are being built around the world [12]. However the best example is the ISOLDE/CERN (Isotope Separator On-Line/European Organization for Nuclear Research) facility at Geneva [13], due to its high diversity, elemental/isotopic purity, and high yields of the radioactive beams. The second requirement, i.e., the adequate sample preparation, will be improved at ISOLDE with the current installation of a post-ac-

celerator for the radioactive ion beams [14]. Such facilities are particularly good to run and develop new applied nuclear techniques. Consequently, the third requirement for the progress of nuclear solid state physics is being developed by an increasing number of collaborations between groups that submit research proposals at ISOLDE/CERN.

In this paper we include a short survey of the principal features of the on-line isotope production at the ISOLDE facility at CERN. A review of the principal nuclear solid state techniques is given and its most recent achievements and perspectives will be presented.

2. The ISOLDE facility at CERN

At ISOLDE radioactive nuclei are produced through spallation, fission or fragmentation reactions in thick targets by the 1 GeV proton beam from the CERN’s PS-Booster [13]. The target products are vaporized and extracted through a transfer line into a selective ion source. The extracted ions are then accelerated to 60 keV and mass (M) separated by analyzing magnets. Two different mass separators are available. The first one, the General Purpose Separator (GPS), provides mass separation with a resolving power $M/\Delta M = 2400$. It is designed to provide up to three simultaneous beams, within a mass range of $\pm 15\%$, delivered to the beam lines in the experimental hall. The second mass separator, the High Resolution Separator (HRS), is equipped with two bending magnets achieving routinely $M/\Delta M = 10,000$. The ISOLDE group develops different types of element-specific target-ion-source combinations that today allow the use of more than 600 radioactive isotopes of about 70 elements [15]. Recently the implementation of chemically selective laser-ion-sources provided new and clean Ag, Ni, and Mn radioactive ion beams [16] and clean beams of Be, Cu, Zn and Cd have also been attained [17].

In order to post-accelerate the radioactive ion beams the approach chosen at ISOLDE preserved the technical status and know-how for the production of radioactive beams, by coupling to the ISOLDE beam line a low cost radio frequency

quadrupole (RFQ) acceleration Linac [14]. Before the Linac, the primary ISOLDE beam is continuously injected into a Penning trap that transforms a continuous (1+) beam to bunches of cooled ions. These are then transferred to an Electron Beam Ion Source (EBIS) in order to achieve a charge to mass ratio larger than 1/4.5. After selection of the desired charge to mass ratio by dipole magnets the ions are gradually accelerated by an RFQ-structure, an interdigital H-type (IH) structure, and a three 7-gap resonators to a final energy of about 2.2 MeV/u. An efficiency of about 10% is expected, essentially given by the EBIS ionization efficiency. For investigations in solid state physics, low energies between 1 and 10 MeV/A can be obtained. For energies below 1 MeV/A, a low charged ion beam (4+) coming out from the EBIS

will be injected through an electrostatic high voltage 0-260 kV acceleration lens [18].

3. Nuclear solid state physics at ISOLDE

The high intensity and wide range of radioactive isotopes have made ISOLDE the ideal facility for solid state physicists using radioactivity in their research. In Fig. 1 the elements are shown for which there are suitable radioactive isotopes for several techniques used in experiments at ISOLDE. Currently, about 35% of all the beam time is dedicated to the solid state physics program, which started during the 1970s with some pioneer experiments, in which the short lived ^{111m}Cd and ^{119}Cd isotopes were implanted into non-cubic metals

H	H Available at ISOLDE																He
Li	Be																
C		C = EC															
N		N = β -NMR															
Na	Mg	M = ME															
C	N	P = PAC															
K	Ca	Sc	Ti	V	Cr	Mn	Fe	Co	Ni	Cu	Zn	Ga	Ge	As	Se	Br	Kr
													C P	C P	C P	P	P
Rb	Sr	Y	Zr	Nb	Mo	Tc	Ru	Rh	Pd	Ag	Cd	In	Sn	Sb	Te	I	Xe
													C P	C P	C M	C M	C
Cs	Ba	La	Hf	Ta	W	Re	Os	Ir	Pt	Au	Hg	Tl	Pb	Bi	Po	At	Rn
P								P		C M	C P				C	C	C
Fr	Ra	Ac															
C	C																
Ce	Pr	Nd	Pm	Sm	Eu	Gd	Tb	Dy	Ho	Er	Tm	Yb	Lu				
					M						C	C	C				
Th	Pa	U	Np	Pu	Am	Cm	Bk	Cf	Es	Fm	Md	No	Lr				

Fig. 1. Periodic table of the elements produced as primary beams at ISOLDE. Shadowed elements have been recently produced with the laser-ion-source. The figure, shows the elements with isotopes which are adequate for several techniques recently used or tested in experiments at ISOLDE. Open symbols represent the probe elements with adequate cascades only obtained by β^- -decay indicated by the left arrow ($\rightarrow$) or obtained by electron capture and β^+ -decay indicated by the right arrow ($\leftarrow$). Currently, other (new) probe nuclei are being tested for application in (e^- , β^- , β^+) EC studies, or to be used by the new "tracer" techniques that only require elements with suitable isotopes of appropriate half-lives.

[19]. Since then the major effort of the solid state physics program has been the study of impurities in semiconductors [20].

Diffusion studies of radioactive tracers are still today an important tool at ISOLDE, being currently used to study the anomalous diffusion of low concentrations of gold in amorphous silicon [21]. In a different type of experiment, the energy spectra of α -particles emitted from the decay of implanted short lived nuclei was shown to be a promising tool for diffusion studies. As an example we mention a test experiment where it has been found that the diffusion coefficients of ^{221}Fr and ^{217}At implanted in potassium are about 1 and 3 orders of magnitude bigger than the K self-diffusion coefficient, respectively [22].

A whole spectrum of more sophisticated techniques which are being used at ISOLDE can essentially be divided into two groups: the well-established “nuclear” and the more recent “tracer” techniques. Nuclear techniques like, e.g., Emission Channeling (EC), ME, PAC and β -NMR spectroscopy, depend critically on the availability of radioactive isotopes with specific decay properties. With these techniques, the radioactive nuclei are used as local probes of the structural or electronic environment in bulk, surfaces, and interfaces of metals and semiconductors.

The EC technique makes use of the fact that charged particles (α , $\beta^\pm$, e^-) emitted from radioactive isotopes in single crystals experience channeling or blocking effects along low-index crystal directions [23]. This leads to an anisotropic particle emission yield from the crystal surface which depends in a characteristic way on the lattice sites occupied by the emitter atoms. In order to decrease the implanted dose and acquisition time, particle detection is done using two-dimensional (2D) position sensitive detectors. One of the specific advantages of this technique is a very low minimum implanted dose, of the order of 10^{12} cm^{-2} , about 2 orders of magnitude below the doses used by conventional channeling based on Rutherford backscattering. As examples, we mention the lattice location studies of Li in elemental and compound semiconductors performed after implantation of the short lived α -emitter ^8Li , to doses even below 10^{12} cm^{-2} [24]. Very recently a new

2D Si pad detector system, that has been developed at CERN for high-energy physics collider experiments [25], has been adapted for use in electron EC experiments [26]. Fig. 2 shows one channeling pattern taken with this new detector along the Si $\langle 111 \rangle$ direction by measuring conversion electrons from the $^{167\text{m}}\text{Er}$ (2.27 s) decay, following room temperature implantation of ^{167}Tm (9.2 d) into FZ p-Si and annealing up to 900°C . The analysis of the $\langle 111 \rangle$, $\langle 110 \rangle$ and $\langle 100 \rangle$ spectra showed that around 93% of the Er atoms are on tetrahedral sites [27]. The ability to use 2D-detectors to detect conversion electrons and $\beta^\pm$ particles finally allows the efficient use of EC probes from almost the entire periodic table. Such detectors are already being envisaged for studies in several subjects [28,29].

The PAC and ME hyperfine techniques represent the most widespread use of radioactive isotopes for solid state research at ISOLDE. These techniques are sensitive to the local environment of the implanted radioactive probe providing information on the defect structure, dynamics, and electronic configuration at the atomic level. PAC and ME rely on the interaction of the nuclear quadrupole or magnetic moments of the probe with the

Fig. 2. Electron emission channeling pattern from $^{167\text{m}}\text{Er}$ (2.27 s) following room temperature implantation of ^{167}Tm into FZ p-Si and annealing at 900°C . Normalized electron emission yields are shown, taken in the vicinity of $\langle 111 \rangle$ axis, from the combined intensity of 150, 198 and 206 keV conversion electrons [27].

electric field gradient (EFG) or magnetic hyperfine field at the probe's location. In particular, the EFG provides information on the local charge density distribution and acts as a fingerprint of a specific defect trapped by the probe nuclei.

Several isotopes are being used in new and exotic ME experiments at ISOLDE. Studies of the structure and electronic states of impurity-vacancy complexes in III-V semiconductors and of Sb doping of Si-based semiconductors such as SiGe alloys are actually carried out at ISOLDE, by using the first excited state of ^{119}Sn , which is obtained either by the decay of the long lived ^{119}Sb (38.2 h) or the short lived ^{119}In (18 m) isotopes [30]. Such studies are being extended to the wide band gap GaN semiconductor in particular, by implanting ^{119}Cs which decays throughout the $^{119}\text{Xe} \rightarrow ^{119}\text{I} \rightarrow ^{119}\text{Te} \rightarrow ^{119}\text{Sb}$ isobaric chain to the Mössbauer ^{119}Sn nucleus. Once the recoil energies (>50 eV) are large enough to place ^{119}Sb in N sites this technique enables the measurement of isomer shifts, i.e., the electron densities, for Sn donors and acceptors on Ga and N sites, respectively [31]. Recently, a new laser source provided pure $^{57}\text{Mn}^+$ beams free of the $^{57}\text{Fe}^+$ contaminant. First studies of interstitial and substitutional Fe in Si could be performed [16] and a new proposal for investigations of deep-level Fe-centers in Si has been presented at ISOLDE [32].

The sensitivity of the PAC technique, contrary to the ME, is essentially temperature independent [6] and a wider range of probe nuclei can be used. At ISOLDE, the PAC technique is being applied to the study of technologically relevant problems of impurity-impurity interactions in semiconductors. The trapping of hydrogen by p-type impurities in III-V and intrinsic semiconductors is being studied using the representative $^{111\text{m}}\text{Cd}$ probe [20]. Due to the breakthrough made by the laser-ion-sources in producing clean Ag^+ beams new experiments that study the hydrogen self-diffusion in III-V semiconductors are being accomplished. After implantation and decay of ^{117}Ag (1.3 m) to ^{117}Cd (2.4 h) the sample is first annealed to recover the implantation defects. Then hydrogen is loaded into the sample, being trapped in the ^{117}Cd acceptor's neighborhood due to Coulomb interaction. The PAC measurement is per-

formed after the ^{117}Cd decays to ^{117}In which is not an impurity in the In-R, $\text{R} = [\text{P}, \text{As}, \text{Sb}]$ III-V compounds, and no longer binds hydrogen by Coulomb interaction. Since the PAC signal senses whether or not hydrogen is located nearby the ^{117}In probe nuclei, the activation energy for the hydrogen self-diffusion can be studied by measuring the PAC spectra as a function of temperature [33]. The recent introduction and development at ISOLDE of the $e^- - \gamma$ PAC technique enlarged significantly the number of probe nuclei and opened up new topics of research [34]. Isotopes with highly converted cascades, inaccessible or inefficient to the traditional $\gamma - \gamma$ PAC technique, such as $^{197\text{m}}\text{Hg}$, $^{199\text{m}}\text{Hg}$ [35] and $^{73}\text{As}/^{73}\text{Ge}$ [36] are now available, providing new possibilities for studying defects in metals, compound semiconductors and n-type impurities in intrinsic semiconductors. Once there is no suitable stable Hg probe nuclei which allows nuclear quadrupole resonance (NQR) measurements to work, the use of the $e^- - \gamma$ technique with the $^{197\text{m}}\text{Hg}$ (24 h) isotope provides a particularly interesting probe to perform research in the field of high T_c -superconductors [29]. In the particular case of $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ (YBCO) compounds, doping studies have shown that the superconducting properties are always degraded, except when doping with Au or Hg, which increase T_c . Fig. 3 shows first PAC results where it has been shown that immediately after implantation of $^{197\text{m}}\text{Hg}$ in high-quality YBCO single crystalline thin films 80% of Hg atoms lie in unique and almost undamaged sites that fully recovered after annealing [29,37]. Furthermore, with the on-line coupling of the $e^- - \gamma$ spectrometer to the ISOLDE beam line new short-lived isotopes are available for PAC experiments, as it has been shown with the first test experiments performed at the $^{127}\text{Ba}/^{127}\text{Cs}$ (12.7 m) and $^{75}\text{Kr}/^{75}\text{Br}$ (4.3 m) decay cascades [38].

Essentially, providing similar information as the ME and PAC techniques, β -NMR works with isotopes of the light alkaline Li, Na and Mg elements. First β -NMR studies of implanted ^8Li in the ZnSe semiconductor are being performed at ISOLDE, with the aim of learning about the Li microscopic symmetry of the implantation sites, site changes, and diffusion behavior [39].

Fig. 3. $e^- \gamma$ PAC experimental, $R(t)$, functions measured after implantation of ^{199m}Hg (top) and after annealing of the ^{197m}Hg implanted single crystal-like YBCO thin film (bottom). Different x-axis scales are due to the different quadrupole moments of the states where the PAC experiments are measured in each isotope [37].

About 25 years were needed since the first less-successful attempt [40], to develop the new “tracer” techniques which combine the standard electrical or optical measurements, commonly used in semiconductor physics, with the use of radioactive isotopes. It was first demonstrated with the identification of the Deep Level Transient Spectroscopy (DLTS) signals from Au and Pt implanted Si, that when the radioactive decay involves the chemical transmutation of the dopant, the electrical properties of the semiconductors will change in time with the characteristic time constant of the radioactive decay [41]. Currently, Hall effect [42], DLTS, Capacitance–Voltage measurements [43] and Photoluminescence (PL) Spectroscopy [44] use radioactive isotopes to overcome the chemical “blindness” of the non-radioactive versions, in studies of the electrical activation of impurities in intrinsic, III–V [45], and II–VI compound semiconductor [46]. Fig. 4 shows PL spectra of undoped and ^{111}In (2.83 d) implanted MBE-grown GaAs taken as a function of the time after doping. The spectra show the increase with time of two PL peaks (on the low energy side of the PL peaks which are due to the sample’s carbon contamination) that clearly could be assigned as due to the

Fig. 4. PL spectra of undoped and ^{111}In doped GaAs successively taken 4 h, 7 h, 12 h, 22 h, 2 d, 4 d, and 9 d after doping. All spectra are normalized to the intensity of the (e,C) peak. In the inset, the height $I_{\text{Cd}}/I_{\text{C}}$ of the (e,Cd) peak in these spectra is shown as a function of time after doping with ^{111}In . The solid line is a fit to the data, which includes the characteristic decay time constant of the $^{111}\text{In}/^{111}\text{Cd}$ nuclear transmutation, as explained in Ref. [45].

$^{111}\text{Cd}_{\text{Ga}}$ acceptors generated by the decay of ^{111}In on Ga sites [45].

New perspectives are open at ISOLDE with the availability of energetic radioactive ion beams. Due to the present beam energy of 60 keV the probe atoms are localized some tens of nanometers below the surface after implantation. For studies of defects in semiconductors, higher energies are often desirable in order to localize the radioactive isotopes deeper in the bulk, that is, outside the surface defects and space charge region. By varying the energy of the ions in the 0.1–10 MeV range, implantation profiles can be produced with a resulting local probe atom concentration of about 10^{15} – 10^{16} cm^{-3} , instead of the current 10^{17} – 10^{18} cm^{-3} . Phenomena that are highly sensitive to the influence of the Fermi level, such as passivation mechanisms due to the trapping of mobile impurities or defects, can be studied in this way. The close vicinity of the surface to the implanted region is also a strong limiting factor when comparing data from different techniques, which are sensitive to different depths.

The future applications of radioactive ion beams are not restricted to semiconductor physics. Radioactive ion beams are currently being applied to study the structure and defects of oxide materials [47,48,29]. The ion beam materials modification is being progressively extended to photonics, open-

ing new ways to the doping of a large variety of photo-sensitive materials. Waveguides and quantum wells are being produced by the implantation of photo-active impurities at selected depths in different substrate materials like LiNbO_3 , and Al_2O_3 [49]. The stability of the dopants in such materials represents today a big challenge, both for technological and fundamental reasons, and represents an additional field of research that can be addressed by the combination of the nuclear techniques with energetic radioactive ion beams.

4. Conclusions

The interface between nuclear and solid state physics has always been a fertile ground for discovering new phenomena and attractive applications. Nowadays the solid state community can profit from a large set of new and powerful nuclear characterization techniques, provided that a dialogue is established with the laboratories that have the know-how of the nuclear techniques. Additionally, two more factors are essential for the success of nuclear solid state research, i.e., a big variety of intense radioactive beams of the appropriate elements, which can only be achieved in on-line mass separators, and adequate sample preparation that will be achieved with the post-acceleration of radioactive ion beams. The combination of these factors will further support the use of complementary techniques, which have been shown in the past to be the most fruitful way to approach the problems in solid state physics. At ISOLDE, the well-established nuclear solid state techniques are being combined with energetic radioactive ion beams, providing new and unique tools for the characterization of materials.

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