Advances in electron emission channeling measurements in semiconductors

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Abstract

This paper reports on recent advances in the application of electron emission channeling to studies of the lattice location of radioactive probe atoms in semiconductors. The introduction of position-sensitive electron detection has resulted in an improvement in the experimental efficiency of this method by two orders of magnitude. Electron emission channeling now allows to carry out detailed lattice location studies, and is especially interesting for cases where conventional ion beam lattice location techniques can not be applied due to a lack of sensitivity. Characteristic features of electron emission channeling with position-sensitive detection are discussed and illustrated by results on the lattice location of Er in Si and GaAs, and on the lattice sites and stability of implanted transition metals in Si.

Keywords: lattice location, position-sensitive detection, ion implantation, Si, GaAs, Er, Cu, transition metals

1. Introduction

Emission channeling, i.e. determining the lattice location of impurity atoms in single crystals by means of channeling and blocking of charged particles emitted in nuclear decay, is a well-established technique among the various nuclear solid state methods at CERN's on-line isotope separator facility ISOLDE. It has been reviewed several times in the past [1-5]. Rather than discussing general aspects of the technique this paper focuses mainly on recent new developments and the current status of the research on the lattice locations of rare earth and transition metal atoms in semiconductors. These examples are well suited to highlight the main benefits of emission channeling compared to conventional ion beam lattice location techniques, which are its superior sensitivity, especially if it is combined with position-sensitive detection techniques, and its applicability to a wide choice of host-impurity systems.

Emission channeling was introduced at ISOLDE in 1985 by Hofsäss and Lindner. Following some initial lattice location studies in metals, the first paper on emission channeling in semiconductors was published in 1986 [6], and described experiments using the conversion electron emitters $^{112\text{m}}\text{In}$ ($t_{1/2}=20.9$ min) and $^{114\text{m}}\text{In}$ ($t_{1/2}=49.5$ d) implanted into Si. Already in the as-implanted state at doses around 10^{12} cm $^{-2}$, indium, which acts as an acceptor in Si, was found with a substitutional fraction of 50%. Following annealing at 923°C, the substitutional In fraction increased to 100% [6]. Earlier lattice location experiments using Rutherford backscattering (RBS) at much higher In doses ($>10^{14}$ cm $^{-2}$) had, even after annealing, only achieved a maximum of 50% substitutional In [7]. Within the ten years from 1986-96, the elemental semiconductors Si, Ge and diamond, the III-V compound semiconductors AlN, GaN, GaP, GaAs, GaSb, InP, InAs and InSb, and the II-VI semiconductors ZnSe, ZnTe and CdTe were studied with various probe atoms from ISOLDE, including ^8Li , ^{73}Se , $^{107}\text{Cd}/^{107\text{m}}\text{Ag}$, $^{109}\text{Cd}/^{109\text{m}}\text{Ag}$, $^{111\text{m}}\text{Cd}$, $^{115\text{m}}\text{Cd}$, ^{111}In , $^{112\text{m}}\text{In}$, $^{114\text{m}}\text{In}$, $^{118}\text{Xe}/^{118}\text{I}$ and $^{118}\text{Te}/^{118}\text{Sb}$ ([1-5, 8-14] and references therein).

While the experiments which used electron or positron emitting probe atoms were somewhat restricted by the fact that no suitable position-sensitive detectors for these particles were available at that time, this obstacle did not apply in the case of the isotope ^8Li ($t_{1/2}=838$ ms). This nucleus decays into an excited state of ^8Be , which immediately breaks up into two α particles with energies around 1.6 MeV. Position-sensitive detectors for MeV α particles were already available, and allowed studies of the lattice sites of ^8Li in all of the semiconductors mentioned above, as a function of implantation temperature and, often also, for different types of doping [1, 2, 5, 8, 9, 12-14]. As a general feature it was found that following implantation at low temperatures Li preferred tetrahedral interstitial sites, while high-temperature implantation lead to the occupancy of substitutional sites in the III-V and II-VI semiconductors, and to bond-centered, substitutional or random sites in Si and Ge. The transition temperature regime was characterized by the onset of Li diffusion. While a number of

other α emitters are of potential interest for emission channeling studies in semiconductors, these probe atoms are not available at ISOLDE, e.g. ^8B or ^{20}Na , or only with insufficient purity, e.g. ^{151}Ho , ^{153}Er or ^{154}Tm .

Two recent experimental developments, however, have considerably improved the possibilities to do lattice location experiments with electron emitters at ISOLDE. The first is the realization of reliable and low-cost position-sensitive detectors for electrons in the 40-1000 keV energy range, which have recently been developed at CERN [15]. This new approach assures that the detection efficiency of electron emission channeling is roughly four orders of magnitude higher than in ion beam studies, and about two orders of magnitude higher than in emission channeling using conventional detection. The second new aspect is the development of laser ionization sources for transition metal isotopes at ISOLDE [16-18]. These ion sources provide for the first time clean beams of radioactive Mn, Cu and Ag ions with sufficient intensity to prepare channeling samples within a short time.

2. Emission channeling with position sensitive electron detectors

2.1 Experimental aspects

The concept of a setup for off-line emission channeling experiments is very simple [1, 5]. The single crystal-line sample is mounted on a goniometer in a high-vacuum chamber. By means of the goniometer the sample can be moved about two or three axes of rotation with a typical angular precision around $0.01\text{-}0.1^\circ$. The sample holder usually includes a heating device which allows for *in situ* thermal annealing of implanted samples. The conventional approach in order to measure the angular-dependent count rate of emitted particles is to rotate the sample step by step at a certain distance from a collimated detector. More conveniently, however, the angular-dependent emission yield is measured by using position-sensitive detectors (PSDs) with large solid angles. An additional advantage of the PSD technique is that the angular accuracy of the goniometer is no longer crucial for the quality of the measurements since no sample movements are needed while taking data. Therefore less precise goniometers can be used and also there is no need for computer-controlled and stepping-motor driven sample rotations.

As has already been mentioned in the introduction, PSDs for MeV α particles have been used at ISOLDE since 1990 for the experiments with the probe atom ^8Li . The search for PSDs suitable for $<1\text{MeV}$ electrons, however, was initially not successful. While multi-wire chambers [19] or double-sided Si strip detectors [20], as they are used, e.g., in high-energy physics or X-ray satellite missions, would have been more than sufficient with respect to position and energy resolution, their price exceeded the budget of a nuclear solid state physics experiment.

Figure 1. Schematic layout of the position-sensitive electron detection system.

superior to strip detectors is required while the position resolution need not be as excellent. It was soon realized that these detectors should also be capable of detecting electrons, though not ideally suited for this purpose due to the rather thick entrance windows and relatively slow counting speeds. The principle of operation of these devices is based on integrating an array of separate detector cells ("pads" or pixels) on a single Si chip and individually contacting them on the surface by a pattern of conducting and insulating layers. Since the signal to noise ratio of individual cells improves with shrinking cell dimensions (due to the decrease in capacitance and leakage current), this allows, to a large extent, a downscaling of both energy and position resolution. In

In 1992 one of CERN's silicon strip detector groups began the development of a new type of position sensitive detector which was to be used in high-energy physics collider experiments and in medical X-ray imaging [15]. The aim was to produce a detector with simple and inexpensive readout electronics for applications where an energy resolution comparable or even su-

contrast to double-sided strip detectors which require accurate processing of both surfaces of a wafer, pad detectors are in principle not very difficult to manufacture. However, their widespread use has been hampered by the high costs of the extensive electronics needed to read out the hundreds, or thousands, of segments. Due to the considerable progress in microelectronics devices, however, integrated multichannel preamplifier circuits have now become available at much lower prices, so that 2-D electron detection has become feasible at moderate cost.

The schematic layout of the detection system which was used for emission channeling experiments with 40-250 keV conversion electrons [21] and <1 MeV β^- particles is shown in Fig. 1. The detectors consist of 30×30 mm² Si wafers of 0.3 mm, 0.5 mm or 1 mm thickness, which are segmented on one side into 22×22 pads, each of 1.3×1.3 mm². The 484 pads are coupled to 4 very-large-scale-integrated (VLSI) preamplifier chips of 128 channels each, which are read out by a digital signal processor (DSP) via an analog to digital converter (ADC) capable of a maximum sampling frequency of 1 MHz. Multiplexed read out of all pads is triggered if the signal on the detector backplane, which is common to all pads, exceeds an externally set lower level threshold. The serial readout procedure, together with the requirement to analyze the signals from all pads, limits the counting speed of the device to 400 events/s. This is sufficient, however, for typical experiments with long-lived radioactive isotopes with a half life above a few hours.

Figure 2. Energy spectrum of ^{167}Tm , recorded with the 1 mm thick Si pad detector; e_K , e_L and e_M indicate the conversion electrons emitted from K, L, and M shells during de-excitation of the two nuclear states in ^{167}Er as shown in Fig. 3.

Figure 2 shows the energy spectrum of the ^{167}Tm decay (cf. sect. 3) which was recorded by the 1 mm thick pad detector at a distance of 285 mm from the sample. The X-ray energy resolution of the detector is found to be 3.1 keV. The somewhat worse energy resolution of 5-6 keV for electrons is due to the energy loss and straggling in the relatively thick detector entrance window (>2 μm), which is also responsible for a shift in electron energy of approximately 8 keV. Concerning the position resolution of the detector, note that besides the pad size of 1.3 mm the lateral straggling of the electrons in the detector is a natural limit. The magnitude of straggling is comparable to the thickness required to completely stop all electrons, which in Si is around 7 μm at 30 keV, 350 μm at 300 keV and 2 mm at 1 MeV.

A critical issue in channeling experiments is the angular resolution $\Delta\theta$, which is usually required to be a few tenth of a degree or better. If σ_d denotes the position resolution during detection, σ_b the position resolution due to the size of the radioactive spot on the sample and d the distance between sample and detector, $\Delta\theta$ is approximately given by

$$\Delta\theta \approx (\sigma_d^2 + \sigma_b^2)^{1/2} / d \quad (1).$$

In the case of conventional detection, sufficient angular resolution can always be achieved at the cost of losing detection efficiency by increasing the distance d between sample and detector and by improving σ_d by using a smaller collimator in front of the detector. Using a PSD, however, the distance between sample and detector is usually defined by the need to cover an angular range of $5-6^\circ$ with a detector of a given size, and σ_d is given by the position resolution of the detector. Therefore, in order to achieve sufficient angular resolution with the pad detectors, the implanted spot should be limited to less than the 1.3 mm size of the pixels, which requires the ISOLDE beams to be collimated during implantation. As a compromise between position resolution and beam transmission, a 1 mm beam spot was chosen, which still allows 30% of the radioactive beam to reach the sample. At ISOLDE the PSDs therefore provide an experimental efficiency which is roughly a factor of 150 higher than conventional detection methods in which beam collimation is not a necessary requirement. At the same angular resolution, a typical PSD sample contains 10-15 times less radioactivity than a conventional one while

still allowing for an order of magnitude more data to be taken within the life time of the implanted isotope. As a consequence of this dramatic increase in efficiency, electron emission channeling experiments can now also take advantage of low-intensity beams at a reasonable number of shifts. For high-intensity beams such as the strongest rare earth elements (yield 10^9 - 10^{10} atoms/ μ A protons) usually a couple of minutes is sufficient to produce a PSD channeling sample.

2.2 Data analysis procedures

In order to clearly identify the lattice positions occupied by the probe atoms and to derive quantitative information on site fractions, it is imperative to compare the experimental patterns to theoretical yields for a variety of different lattice sites by means of a fitting procedure. The information which is contained in the channeling patterns measured by a PSD can only be fully exploited by comparing to theoretical yields which cover the same range of angles. The introduction of position-sensitive electron detection hence went along with an upgrade from simulating and fitting one-dimensional scans to simulating and fitting entire two-dimensional patterns. While the necessary fitting routines had already been developed for the analysis of ^8Li α emission channeling [13], the need to optimize the electron channeling computer simulations soon became obvious.

The theoretical framework for the description of electron channeling in single crystals was laid out already in the 1980s by Andersen et al [22] and is closely related to the "many-beam" theory of electron microscopy. The basic idea is that high-energy electrons moving under small angles from a crystal axis effectively sense only a continuum potential which is averaged along the axis. The electron wave function may then be split into an axial, relativistic component, and a transverse component which is given by the solution of a non-relativistic Schrödinger equation in a two-dimensional periodic potential. The transverse electron eigenstates are therefore Bloch waves, i.e. plane waves multiplied by a function with the periodicity of the two-dimensional lattice. Electron motion is then described by the superposition of all eigenstates which results from matching to the boundary condition of a plane wave at the crystal surface. Numerical treatment of this problem is possible by means of approximating the continuum potential and the transverse electron wave functions by suitable numbers of Fourier components. The continuum potentials are conveniently derived from Doyle-Turner potentials [23, 24], which are screened Coulomb potentials obtained from a fit to the results of first-principles Hartree-Fock calculations of atomic electron densities. Doyle-Turner potentials are especially suited for electron diffraction theory, and they are widely used in electron microscopy or to describe reflection high energy electron diffraction (RHEED) [24].

Figure 3. Simplified decay scheme of ^{167}Tm (some low-intensity EC transitions are not shown); f_K , f_L , and f_M are conversion electron intensities with reference to the decay of ^{167}Tm .

Hofsäss and Lindner developed a computer program, called "MANYBEAM", which adapted the theoretical concepts described above to the specific situation of emission channeling [1]. The MANYBEAM code was found to accurately describe electron emission channeling effects in a variety of cases [1-4], provided of course that a sufficient number of Fourier components, which increases with electron energy and nuclear charge of the crystal atoms, were considered. For instance, in order to calculate the yields from 150 keV electrons in Si within a convergence precision better than 0.5%, at least "16-beam" simulations are needed, i.e. the two-dimensional potential must be represented by at least 33×33 Fourier components. Note that in a 16-beam simulation the resulting electron wave function is given by a superposition of 289 eigenstate matrices which are (17×17) component symmetric and real in the case of two-dimensional lattices with both x and y mirror symmetry, and complex Hermitean in all other cases.

Typically, electron yields are calculated for an angular range of 0° to 3° from the axis in steps of 0.05° , i.e. considering 3721 angles in case of a fourfold symmetric axis. For every angle the eigenvalue problem must be solved and the electron flux density at 40-60 different lattice sites calculated as a function of depth. In particular, if a large number of beams or if complex wave functions are needed, as in the case of GaAs or GaN, or if many different electron energies must be considered, e.g., for β^- emitters with continuous energy spectra, this represents an extremely heavy computing task. However, fortunately it was recognized that the most time critical parts of the MANYBEAM source code could be replaced by highly optimized, sometimes CPU specific manipulation routines for symmetric or Hermitean matrices. It was thus possible to speed up the execution of

MANYBEAM on most computers by roughly a factor of 10. A typical 16-beam Si simulation now runs on high-end workstations within a couple of days and even on ordinary PCs within one to two weeks.

The simulations result in site-characteristic two-dimensional patterns of electron emission probability, $\chi_{\text{theo}}(\theta, \phi)$, where θ and ϕ denote polar and azimuthal angles from the axis. The patterns for different electron energies are summed according to the conversion electron branching ratios or the intensity distribution of the β^- spectra, and smoothed using a Gaussian of $\sigma = 0.06^\circ$ to account for that part of the experimental angular resolution which is due to the 1 mm beam spot on the sample. The size and shape of the detector pads, however, is taken into account during fitting by averaging over the simulated yield falling within the angular range $(0.26^\circ \times 0.26^\circ)$ of one pad. Theoretical emission patterns are fitted to the experimental yields, χ_{ex} , according to

$$\chi_{\text{ex}}(\theta, \phi) = S [f_1 \chi_{\text{theo},1}(\theta, \phi) + f_2 \chi_{\text{theo},2}(\theta, \phi) + f_3 \chi_{\text{theo},3}(\theta, \phi) + 1 - f_1 - f_2 - f_3] \quad (2),$$

where S is a scaling factor common to all angles in one pattern, and f_1 , f_2 and f_3 denote the fractions of emitter atoms on up to three different lattice sites. The random fraction, $f_R = 1 - (f_1 + f_2 + f_3)$, accounts for emitter atoms which cause negligible anisotropies in emission yield, i.e. which are located on sites of very low crystal symmetry or in heavily damaged or amorphous surroundings. Up to seven fitting parameters, S , f_1 , f_2 , f_3 , x_0 , y_0 and ϕ_0 , may be simultaneously optimized using non-linear least square fitting routines. While the parameters S , x_0 , y_0 and ϕ_0 are always allowed to vary in order to provide correct normalization of the experimental spectra and to achieve optimum translational and azimuthal orientation with respect to the detector, usually only one or two different site fractions, f_1 and f_2 , are considered.

3. Lattice location of rare earth atoms

Rare earth doped semiconductors are of considerable interest as optoelectronic devices [25]. Among the various

Figure 4. (a)-(c): channeling patterns from $^{167\text{m}}\text{Er}$ (2.28 s) following room temperature implantation of ^{167}Tm into p -Si FZ and after finishing an annealing sequence to 900°C . Shown are normalized electron emission yields from the combined intensity of 150, 198 and 206 keV electrons in the vicinity of $\langle 111 \rangle$, $\langle 100 \rangle$ and $\langle 110 \rangle$ directions. (d)-(f): best fits of simulated patterns to the experimental yields, corresponding to 86%, 95% and 99% of emitter atoms on sites which are displaced by $0.43 \text{ \AA}$ from the T site (from Ref. [32]).

rare earth elements, Er is especially attractive since its atomic transition at $1.54 \mu\text{m}$ matches the absorption minimum of SiO_2 . Possible applications include infrared light emitting diodes as a direct link between semiconductor and glass fiber technology or for optical on-chip communication [26]. The optical activity of Er in semiconductors is closely related to its lattice site and immediate surroundings. For instance, in Si the formation of complexes between O and Er can increase the room temperature luminescence yield by orders of magnitude [26, 27]. This is evident from a comparison of the Er luminescence in float zone (FZ) Si, where O is present only at concentrations around 10^{15} - 10^{16} cm^{-3} , with Czochralski (CZ) Si ($[\text{O}] \approx 10^{17}$ - 10^{18} cm^{-3}) or O co-implanted Si. Photoluminescence (PL) spectroscopy studies [28] suggest that, while a PL center with cubic symmetry may be attributed to isolated interstitial Er, Er-O complexes produce several PL lines with less than cubic symmetry, the most intense Er-O center showing axial symmetry. The existence of tetrahedral interstitial (T) Er would be also in agreement with theoretical studies, which predict that T sites are the most stable sites for isolated Er atoms in Si [29]. RBS channeling experiments, however, had only suggested substitutional (S) [30] or hexagonal interstitial (H) Er [31].

In the following the emission channeling studies [32-37] are reviewed which gave the first direct and unambiguous evidence for tetrahedral interstitial Er in Si. In order to

probe possible Er sites, the isotope ^{167}Tm ($t_{1/2}=9.25$ d) was implanted. ^{167}Tm decays into two excited states of ^{167}Er with half lives of 1.5 ns and 2.28 s, respectively (Fig. 3). While the 1.5 ns state emits L and M conversion electrons of 48 and 56 keV energy, the decay of the 2.28 s state ^{167m}Er is accompanied by K, L and M electrons of 150, 199 and 206 keV. Both types of electrons can be used for lattice location purposes, and they probe the lattice sites of ^{167}Er during different time windows following the decay of ^{167}Tm .

Figures 4 (a)-(c) show the normalized emission yield of conversion electrons from the 2.28 s state ^{167m}Er in a *p*-Si FZ crystal (implanted dose $4.8 \times 10^{13} \text{ cm}^{-2}$) at the end of an isochronal annealing sequence from room temperature up to 900°C (10 min, steps of 100°C). The electron channeling effects in axial $\langle 100 \rangle$ and $\langle 111 \rangle$ and planar $\{110\}$, $\{100\}$ and $\{211\}$, in combination with the decrease in yield along axial $\langle 110 \rangle$, and planar $\{111\}$ and $\{311\}$, give already clear evidence that the majority of ^{167m}Er must reside close to tetrahedral interstitial sites. The best fit results [Figures 4 (d)-(f)] were thus obtained for 93(7)% of tetrahedral interstitial Er with a root mean square (rms) displacement of $d=0.43(8)$ Å from the T site [32]. The emission channeling effects of the 48 keV and 56 keV electrons originating from the 1.5 ns state in ^{167}Er were also fitted and resulted in the same site preference as discussed above, i.e. around 100% near-T with $d=0.44(10)$ Å. This directly shows that practically all ^{167}Er atoms already occupy the near-T sites within less than 1 ns after the ^{167}Tm decay and stay there for at least a couple of seconds.

However, the issue of a possible influence of nuclear decay on the ^{167}Er lattice sites must be addressed. Since the neutrino recoil energy imparted to the ^{167}Er nucleus is only 0.7-0.8 eV, it is very likely that ^{167}Er has inherited its lattice site from ^{167}Tm . On the other hand, the electron capture decay leaves the Er atom in a highly excited state which might result in metastable sites. Both arguments put into question whether the lattice locations observed by conversion electron emission channeling are actually thermally stable Er sites. Fortunately, these questions were answered by *in situ* measurements at 900°C . At this temperature ^{167m}Er is already able to make several diffusion steps during its 2.28 s half life, but the emission channeling results showed that still the same lattice sites were preferred [34]. One feature of the nuclear decay remains, however. While the lattice location is uniquely determined for Er, the thermal processing always acts on a mixture of Tm and Er. The chemical similarity of both rare earth elements, however, allows to draw conclusions on the behaviour of Er in Si as such. Still, it should be kept in mind that, if in the following sections Er concentrations, Er doses, Er_n complexes etc. are discussed, this actually refers to a mixture of Tm and Er.

Figure 5. Fractions of ^{167m}Er on near-T sites obtained during isochronal annealing sequences (10 min, measurements at 20°C) for FZ (a) and CZ Si samples (b), and isothermal annealing sequences at 900°C for FZ (c) and CZ Si samples (d). Sample CZ 4, indicated by §, was annealed at 950°C instead of 900°C (from Ref. [35]).

So far 4 FZ and 12 CZ Si single crystals of different doping character have been investigated [35-37] with implanted Tm+Er doses between $4 \times 10^{12} \text{ cm}^{-2}$ and $5 \times 10^{13} \text{ cm}^{-2}$, corresponding to peak concentrations of $2 \times 10^{18} \text{ cm}^{-3}$ and $2.4 \times 10^{19} \text{ cm}^{-3}$. Figures 5(a) and 5(b) summarize the fractions of ^{167m}Er on the near-T sites derived from isochronal annealing sequences (10 min) up to 850°C . In the as-implanted state the fraction on the

near-T sites decreases with increasing implantation dose due to the accumulation of damage and the onset of amorphization. Upon annealing to 550-600°C solid phase epitaxial regrowth, the main annealing step of implanted Si, removes most of the implantation damage and the near-T Er fractions reach 80-100%.

Figure 6. The data points show the percentage of ^{167m}Er atoms which are left on near-T sites in Si as a function of isothermal annealing time at 900°C, relative to the near-T fractions obtained after annealing at 600°C.

(a): CZ Si. Solid lines: fractions of Er remaining outside $\text{Er}_n\text{O}_{m\geq 1}$ clusters calculated for the four different Tm+Er peak concentrations according to the model in the text; dashed lines: the same for $\text{Er}_n\text{O}_{m\geq 2}$ clusters. Er.

(b): FZ samples. Dashed line: fraction of Er remaining in the sample under the assumption that diffusing Er is not interacting and only trapped at the surface; solid lines: fractions of Er remaining outside Er_n clusters for the highest and lowest implantation dose according to the model described in the text.

While up to 750°C FZ and CZ Si samples implanted with the same doses show the same behaviour, at higher annealing temperatures, especially at 900°C, pronounced differences are evident. The near-T fractions in CZ Si decrease rapidly for isothermal annealing at 900°C [Fig. 5(d)], but only prolonged annealing of the order of hours is able to reduce them in FZ material [Fig. 5(c)]. Furthermore, in CZ Si the rate of decrease is anti-correlated with the implanted dose, i.e. the stability on near-T sites is higher for samples implanted with a higher dose of Er. In FZ Si the stability of near-T Er was highest for the sample implanted with the lowest dose. An explanation for this behaviour, especially for the dose dependence in CZ Si, would be the formation of Er-oxide in CZ Si and Er-silicide precipitates in FZ Si. In order to compare the remaining near-T fractions in the channeling experiments to possible scenarios of Er-O and Er-Er clustering, a numerical capture model was developed on the basis of simple diffusion and reaction kinetics. Both Er and O diffuse at 900°C and, assuming that they interact with each other within certain capture radii, coupled differential equations may be set up for the concentrations $C_{\text{Er}(n)\text{O}(m)}$ of Er_nO_m complexes. These equations are then solved by the finite differences method. This means that, starting from an initially constant O concentration and a Gaussian depth profile of Er, the concentrations $C_{\text{Er}(n)\text{O}(m)}$ of all desired Er- and O-containing defects are updated within finite time intervals Δt and depth intervals Δx . The diffusion coefficients of erbium ($\approx 10^{-15} \text{ cm}^2\text{s}^{-1}$ at 900°C) [27] and oxygen [$0.13 \text{ cm}^2\text{s}^{-1} \times \exp(-2.53\text{eV}/kT)$, corresponding to $1.8 \times 10^{-12} \text{ cm}^2\text{s}^{-1}$ at 900°C] [38] are taken from the literature, while all complexes are assumed to be immobile. A detailed description of the model can be found in Ref. [36].

Figure 6(a) compares some model predictions for CZ Si to the experimental data. All of the 6 CZ Si samples shown had a well-characterized O concentration of $6.5\text{-}6.6 \times 10^{17} \text{ cm}^{-3}$. The fraction of Er which is removed from near-T sites is clearly correlated to the amount of O which can be delivered by the bulk of the sample during the time of annealing. According to the detailed model calculations, mainly Er_nO_m clusters with a few Er atoms ($n=1,2,3$) should be formed under such circumstances. For all six different doses the experimental fractions of Er on near-T sites lie very close to the expected values for free Er atoms plus Er within Er_n

clusters, i.e. the Er fractions remaining outside of any $\text{Er}_n\text{O}_{m \geq 1}$ complexes. This indicates that only one O atom per cluster is required to remove an Er atom from its near-T site [37].

In the case of FZ Si with typical oxygen concentrations of 10^{15} - 10^{16} cm^{-3} , the capture model predicts that the formation of Er_nO_m complexes is negligible. However, it is still unclear what causes the Er atoms to leave their near-T sites. If one completely neglects any interactions of Er and considers the surface as a perfect sink, the dashed line in Fig. 6(b) shows the fraction of Er remaining in the bulk of the sample. On the other hand, assuming that Er forms stable and immobile Er_n clusters, the two solid lines in Fig. 1(a) represent the fraction of free Er remaining outside of any clusters for the highest ($2.4 \times 10^{19} \text{ cm}^{-3}$) and lowest ($2.7 \times 10^{18} \text{ cm}^{-3}$) Er peak concentrations, respectively. Note that both for the highest and lowest dose this process would result in approximately 63% of Er in Er_2 , 25% in Er_3 , 6% in Er_4 and only 2% in higher order clusters. The experimental results therefore show that either these clusters are not formed at all or that Er still occupies the near-T sites within the clusters. In the latter case, a possible scenario would be that the small clusters dissolve again on time scales around 1000-10000 s, resulting in the formation of larger clusters and finally precipitation.

Figure 7. (a)-(c): Channeling patterns from $^{167\text{m}}\text{Er}$ following room temperature implantation of ^{167}Tm into $n\text{-GaAs:Te}$ and annealing at 100°C , 200°C and 300°C for 10 min. The mirror-asymmetry of the <110> pattern, clearly visible, e.g., along the $\{110\}$ plane, is direct evidence that Er prefers a specific sublattice. (d)-(f): Best fits of simulated patterns to the experimental yields, corresponding to the Er fractions on S_{Ga} and T_{As} sites given in the text (from Ref. [48]).

The samples used in the emission channeling experiments were two <100> cut $n\text{-GaAs:Te}$ single crystals ($0.6\text{-}2 \times 10^{-3} \Omega\text{cm}$, $n=1\text{-}5 \times 10^{18} \text{ cm}^{-3}$) which were implanted at 60 keV under 7° from the surface normal with doses of $6 \times 10^{12} \text{ cm}^{-2}$ (sample A) and $3 \times 10^{13} \text{ cm}^{-2}$ (sample B). These parameters correspond to $[\text{Tm}+\text{Er}]$ peak concentrations of $3 \times 10^{18} \text{ cm}^{-3}$ (A) and $1.5 \times 10^{19} \text{ cm}^{-3}$ (B) at a depth of $198 \text{ \AA}$ (FWHM $184 \text{ \AA}$). Figures 7(a)-(c) show the normalized $^{167\text{m}}\text{Er}$ conversion electron emission yields around the <111>, <100> and <110> direc-

The behaviour of Er in the III-V semiconductor GaAs is different from Si. Theoretical investigations suggest that isolated Er prefers substitutional Ga sites (S_{Ga}) and that even the interaction with additional native defects such as Ga_i , As_i , or V_{As} is hardly able to remove it from this site [39]. Er on both tetrahedral interstitial sites is considered to be metastable, but the T_{As} sites should be more stable than the T_{Ga} sites. On the experimental side, a number of RBS lattice location studies revealed the following trends. In general, samples doped with Er during metalorganic chemical vapour deposition (MOCVD) [40-42] or molecular beam epitaxy (MBE) [43-45] showed the majority of Er in near-tetrahedral interstitial locations with respect to the GaAs lattice irrespective of Er concentration. In implanted samples, Er was found to prefer substitutional sites at concentrations around 10^{19} cm^{-3} but tetrahedral interstitial around 10^{20} cm^{-3} [46, 47]. The Er atoms on T sites were supposed to be located either in complexes with carbon or in small ErAs precipitates. In none of the RBS studies, however, it was possible to discriminate between the Ga and the As sublattice. In contrast, electron emission channeling allows to extend the Er lattice location experiments to lower doses and, at the same time, to pinpoint the sublattice [47], which will be discussed in the following.

tions of sample A following an annealing sequence to 300°C. Pronounced emission channeling effects are visible along all axial and planar directions, which is clear evidence that the majority of $^{167\text{m}}\text{Er}$ occupies substitutional sites. Extracting reliable lattice location information for the III-V compound GaAs requires to consider the characteristic properties of the underlying zincblende structure. For instance, the $\langle 111 \rangle$ patterns do not allow to discriminate between S and T sites; in addition both the Ga and the As sublattice show the same projection along this direction. Along $\langle 100 \rangle$ directions S_{Ga} and T_{As} sites lie within pure Ga rows while S_{As} and T_{Ga} are located within pure As rows. By T_{As} we denote those T sites which have 4 nearest As neighbours in $\langle 111 \rangle$ and 6 next-nearest Ga neighbours in $\langle 100 \rangle$ directions. In principle the $\langle 100 \rangle$ axis could therefore allow to discriminate between $(S_{\text{Ga}}+T_{\text{As}})$ and $(S_{\text{As}}+T_{\text{Ga}})$. However, the nuclear charges of Ga and As are so similar that in practice it is impossible to distinguish the $\langle 100 \rangle$ Ga and As rows reliably. Finally, the $\langle 110 \rangle$ patterns allow to distinguish between all various interstitial sites and between both sublattices. Rather than being a consequence of the difference in nuclear charges along the $\langle 110 \rangle$ Ga or As rows, the sublattice sensitivity is a structural effect related to the mirror-asymmetry of the $\langle 110 \rangle$ directions of the GaAs lattice. Hence Er preferring sites in one specific sublattice, e.g., S_{Ga} , causes an asymmetry in the $\langle 110 \rangle$ patterns. On the other hand, if the Er would be equally distributed among both sublattices, the electron yield around the $\langle 110 \rangle$ direction should be almost symmetric with respect to mirroring at the $\{100\}$ plane, which is not the case [Fig. 7(c)]. Correspondingly, best overall fits were obtained for 68(3)% of Er on S_{Ga} with $u_1=0.16$ Å, 16(3)% on T_{As} , and 19(3)% on random (R) sites. Possible Er fractions on S_{As} or T_{Ga} are less than 5%.

Figure 8. Fractions of $^{167\text{m}}\text{Er}$ on S_{Ga} , T_{As} and R sites as function of isochronal (10 min) annealing temperature for samples A (a) and B (b). All measurements were done at room temperature, except the open data points for sample A, which were measured at 500°C following the 600°C anneal step (from Ref. [48]).

Figure 8 summarizes the results of the annealing sequences for the two samples. In both cases clear channeling effects were already visible directly following room temperature implantation, corresponding to around 38% of Er on S_{Ga} sites in the low-dose sample A, and 13% in the higher-dose sample B. The remaining Er fraction occupied random sites, i.e. sites in disordered surroundings which are responsible for an isotropic electron emission yield. In sample A the channeling effects improved markedly following annealing at 200°C, resulting more or less in the Er fractions on S_{Ga} and T_{As} sites discussed above. On the other hand, in sample B the recovery step was shifted to higher temperatures and a larger amount of Er (50%) remained on R sites. This behaviour indicates incomplete structural recovery of the radiation damage in this higher-dose implanted sample. In between the annealing steps at 600°C and 650°C, sample A was heated again at 500°C for 2 h in order to measure *in situ* the emission channeling effects at this temperature. The results were essentially the same as measuring at room temperature following the 600°C anneal and have been included in Fig. 8. This shows that the S_{Ga} sites are thermally stable at 500°C during the life time of the excited $^{167\text{m}}\text{Er}$ state and not only metastable positions which are populated as a consequence of the ^{167}Tm decay.

In both samples a smaller part of Er was found on the T_{As} sites and this fraction increased with increasing annealing temperature at the expense of the S_{Ga} sites. Er on T_{As} sites cannot be explained by the formation of epitaxial ErAs precipitates (the Er atoms at the surface of the precipitates are supposed to be located on T_{Ga}

sites [47]), and also the probability of forming such clusters should be higher in the higher dose sample B. The increasing occupation of T_{As} sites is therefore not well understood at the moment. The vanishing of the channeling effects following annealing above 600°C is not surprising. The samples were annealed capless in vacuum $<10^{-5}$ mbar and it has been reported in the literature that heating of GaAs above 500°C without As overpressure or capping causes excessive As losses within the first few hundred Å and hence sample degradation [49].

The results on Er in GaAs can be compared to the lattice location of Pr [50], and Er and Tm [51] in the wide-bandgap III-V semiconductor GaN. Similar to Er in GaAs, ^{143}Pr , $^{167\text{m}}\text{Er}$, $^{169*}\text{Tm}$ were found to preferentially occupy substitutional sites in hexagonal GaN. Two-dimensional channeling patterns from ^{143}Pr were recorded around the [0001], [1102], [1101] and [2113] directions. The patterns from the [1102], [1101] and [2113] directions, which have separated rows of Ga and N, clearly proved the preference of Pr for substitutional Ga sites. The emission channeling experiments with $^{167\text{m}}\text{Er}$, $^{169*}\text{Tm}$, on the other hand, did not use position-sensitive detection and hence less data are available, which is one of the reasons that the sublattice of Er and Tm could not be determined.

4. Lattice location of transition metal atoms

Figure 9. (a), (b), (c): β^- channeling patterns following room temperature implantation of $3.8 \times 10^{12} \text{ cm}^{-2}$ of ^{67}Cu into $n\text{-Si:P FZ}$ and annealing at 200°C. Shown are normalized emission yields from the integral β^- intensity in the vicinity of $\langle 111 \rangle$, $\langle 100 \rangle$ and $\langle 110 \rangle$ directions. (d), (e) and (f) are best fits of simulated patterns to the experimental yields, corresponding to 70%, 62%, and 76% of emitter atoms on sites which are displaced by 0.50 Å from the S site towards the BC site (from Ref. [57]).

Despite their technological importance as contaminants and deep electronic centers, the lattice sites of transition metal atoms (TMs) in semiconductors are not well studied [52-54]. This is partly due to the fact that in most semiconductors TMs are only stable at such small concentrations that conventional lattice location techniques by means of ion beam channeling cannot be applied. The most recent emission channeling experiments at ISOLDE therefore aim at the lattice location of Fe, Cu and Ag in semiconductors. The probe atoms which are used for that purpose are the β^- emitters ^{59}Fe (44.6 d, $\beta^- < 0.461 \text{ MeV}$), ^{67}Cu (61.9 h, $\beta^- < 0.577 \text{ MeV}$), and ^{111}Ag (7.45 d, $\beta^- < 1.035 \text{ MeV}$). While ^{67}Cu and ^{111}Ag are directly available from the Cu and Ag laser ion sources, ^{59}Fe may be implanted via short-lived ^{59}Mn (4.6 s) by means of the Mn laser ion source. The β^- decay of ^{59}Mn to ^{59}Fe (endpoint energies 4.4 MeV and 4.8 MeV) is accompanied by a nuclear recoil around 200 eV, which assures that ^{59}Fe is reimplanted and its lattice position is not influenced by the previous site of ^{59}Mn .

1998 was the first year for the transition metal lattice location experiments to take beam at ISOLDE. From the initial results it could already be shown that Fe, Cu and Ag are all incorporated on or near substitutional sites following room temperature implantation into Si, which represents the first successful direct lattice location studies for these elements in Si. Figure 9 shows as an example the angular-dependent β^- emission yield around the $\langle 111 \rangle$, $\langle 100 \rangle$ and $\langle 110 \rangle$ axes of a Si single crystal implanted with ^{67}Cu at a dose of $3.8 \times 10^{12} \text{ cm}^{-2}$. The channeling effects along all major crystal axes and the closest packed planes, $\{111\}$ and $\{110\}$, are clear evidence that the majority of implanted Cu is located close to substitutional lattice sites. While $\{100\}$ planar channeling in Si is usually not very pronounced (a few per cent above unity), in this case even an electron emission yield below unity was observed along the $\{100\}$ planes. This gives evidence that the Cu atoms exhibit a significant displacement from ideal substitutional lattice sites. A detailed investigation of the emission channeling data [55, 56] has shown that the most probable Cu positions are sites of trigonal symmetry, displaced around $0.50(8) \text{ \AA}$ along the $\langle 111 \rangle$ directions towards the bond center positions.

Annealing of the implanted Si samples showed that substitutional Cu, Fe and Ag are stable up to around 200°C , 400°C and 500°C , respectively. At temperatures above the stability limit, the TMs leave the substitutional sites and start to diffuse throughout the sample. These results are also technologically interesting for the following reasons. Fe, Ni and Cu are relatively abundant contaminants in the processing of Si integrated circuits (ICs). Via interstitial diffusion these metals may even at room temperature penetrate whole wafers within days or even hours, which degrades the electrical properties due to the introduction of deep levels or the passivation of dopants. The miniaturization of devices therefore requires one to keep their concentration at progressively smaller and smaller values, currently below 10^{11} cm^{-3} . Besides isolating them from the Si wafer production and processing, one therefore adopted the additional strategy of gettering the remaining transition metal impurities outside the active region of devices. One possibility of gettering is trapping at deliberately created radiation damage [57]. In the case of Cu, which, since a few years, is increasingly used for electrical interconnects on top of advanced chips [58], and hence has entered IC production at one of its last processing steps, the issue of diffusion barriers and gettering will remain crucial. The current channeling experiments clearly demonstrate that the room temperature interstitial diffusion is entirely suppressed once the TMs occupy substitutional sites, which in turn strongly suggests that the formation of substitutional transition metals is involved in the radiation damage gettering mechanism.

In the future the TM lattice location experiments will be extended from Si to Ge and diamond and to III-V semiconductors such as GaAs, GaN, or InP. Fe represents the most common TM contamination in GaN [59], while in the case of InP intentional Fe doping is beneficially used to convert from low-resistivity to semi-insulating material. Recently, the implantation of Fe in InP has gained increasing interest due to its potential for creating buried semi-insulating layers [60].

5. Conclusions

Due both to the introduction of position-sensitive detectors and substantial progress in its data analysis procedures, electron emission channeling has reached a high degree of maturity as a lattice location technique. Its main merits lie in the study of systems which cannot, or only with difficulty, be investigated by conventional ion beam lattice location methods, or in measuring below the dose range accessible to these methods. Besides, the superior quality of the emission channeling data can often give significant new information. Three typical examples have been presented.

In the case of Er-doped Si, electron emission channeling provided the first direct evidence that tetrahedral interstitial sites are the most stable Er sites. These results are in accordance with theoretical predictions and put the interpretation of two previous higher dose RBS studies into question. Due to the high efficiency, it was also possible to study the lattice sites of Er in detail as a function of annealing temperature and implanted dose in various different types of Si, including *p*- and *n*-doped material, as well as oxygen-lean FZ and oxygen-rich CZ Si. The combination of the experimental results with a theoretical model for Er and O diffusion and capture lead to the conclusion that the trapping of diffusing O is responsible for the removal of Er from near-tetrahedral lattice sites, leading to its incorporation on random lattice sites.

For Er-implanted GaAs, emission channeling experiments were done at a dose which is comparable to previous low-dose RBS studies but also at ten times lower dose. In the former case both methods gave similar results. However, due to the superior quality of the two-dimensional emission channeling patterns in combination with simulation results, it was also possible to pinpoint the sublattice preference of Er. In agreement with theoretical predictions, the major fraction of Er was found on substitutional Ga sites and a smaller fraction on tetrahedral interstitial T_{As} sites.

The study of the lattice sites of transition metals in semiconductors represents the most recent type of channeling experiments at ISOLDE and focuses on systems which are hardly accessible to conventional ion beam methods. Results on Fe, Cu and Ag in Si are already available. While the Fe and Ag experiments are

currently analyzed in detail, it is already obvious from the raw data that all three transition metals end up on or near substitutional lattice sites following implantation into Si and that their stability on these sites is high.

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