

Electron emission channeling spectroscopy using X-ray CCD detectors

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Abstract

The lattice sites of impurity atoms in crystals can be determined using the emission channeling (EC) technique. This method provides also information on the diffusion behavior and the defect interaction of impurities. In the EC technique, crystals are doped with radioactive impurity atoms and the anisotropic emission distribution of decay particles, preferably conversion electrons with typical energies of the order of 100 keV, are measured around different crystallographic directions. The measurement of EC spectra requires the energy- and angle-resolved detection of decay electrons with an angular resolution of typically 0.1° . We present first electron EC measurements using an energy- and position-sensitive CCD detector originally developed for imaging soft X-rays. We have prepared a sample by implanting radioactive ^{111}In ($t_{1/2} = 2.8$ days) into Si (100) and subsequent annealing to 1173 K. In this sample In occupies substitutional sites and channeling effects, i.e. an enhanced emission yield, along all major crystallographic axes and planes is expected. The $\langle 100 \rangle$ axial electron emission distribution of 147 keV and 219 keV conversion electrons were recorded using a 64×200 pixel CCD for an angular range of $\pm 2.9^\circ$ vertically and $\pm 8.5^\circ$ horizontally. The CCD is an excellent detector for conversion electrons with extremely low noise and an outstanding energy resolution of about 3 keV FWHM at 219 keV. Future applications of CCD detectors for conversion electron spectroscopy are outlined.

Keywords: pn-CCD; electron detector; emission channeling; conversion electron spectroscopy; lattice location;

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1. Introduction

The lattice sites of impurity atoms in crystalline solids can be determined with high precision and high sensitivity using the emission channeling (EC) technique. In comparison to the commonly used ion channeling technique, EC allows lattice location studies at extremely low impurity concentrations of about $10^{15} - 10^{18} \text{ cm}^{-3}$, corresponding to a fluence of $10^{10} - 10^{13} \text{ ions/cm}^2$ in the case of impurities introduced by ion implantation. The fundamentals and applications of emission channeling are described in several review articles [1,2].

For emission channeling studies, crystalline samples are doped with radioactive impurities and the anisotropic emission distribution of energetic decay particles, such as β -particles, conversion electrons or α -particles are measured for different crystallographic directions. From an analysis of these emission distributions one can extract quantitative information on the impurity lattice site distribution, the presence of implantation damage and other defects, the impurity-defect interaction and the diffusion behavior of the impurities. In particular for the characterization of ion-implanted semiconductor materials, EC is an ideal technique complementary to conventional techniques such as photoluminescence spectroscopy, deep level transient spectroscopy DLTS and Hall effect measurements.

The measurement of the emission distributions requires energy resolving particle detectors to scan an angular range of typically $\pm 5^\circ$ with about 0.1° resolution around different crystallographic directions of the sample. Thus, energy- and position-sensitive particle detectors are the best choice.

Until recently, suited detectors for position- and energy-resolved detection of energetic electrons were not available. The electron emission distributions were recorded by scanning the desired angular regime using a collimated semiconductor detector and rotating the sample using a precision goniometer. Thus, EC studies with electron emitting impurities were time consuming and often of limited accuracy due to poor counting statistics.

For the detection of α -particles there exist position- and energy-resolving semiconductor detectors

based on the charge-division principle [3]. These detectors could be used very efficiently for a number of EC studies using the α -emitting isotopes ^8Li and ^{221}Fr [1,2]. However, as discussed later on, they are not suitable for electron detection.

Recently, Wahl et al. performed EC studies using position sensitive silicon pad detectors for energetic electrons [4]. Those detectors were proto-types, originally developed at CERN as hybrid photon detectors for soft X-rays [5].

In this paper we present first results on electron channeling measurements using an X-ray CCD detector [6]. We will demonstrate that imaging of energetic electrons with excellent energy resolution is possible with such a detector.

2. A brief introduction to emission channeling

The EC technique was routinely applied for impurity lattice location studies by Hofsäuss und Lindner since 1985, utilizing the isotope separator facilities for radioactive ion implantation [7]. For EC experiments, crystalline samples are doped with radioactive impurities typically by ion implantation at isotope separator facilities like ISOLDE/CERN [8] or ion implanters dedicated to radioactive ion beam implantation like the Göttingen IONAS facility. ISOLDE provides a variety of different radioactive isotopes suitable for EC studies. With the exception of the α -emitting isotopes ^8Li and ^{221}Fr all other isotopes used for EC studies so far emit β -particles or conversion electrons. The position- and energy-resolved detection of energetic electrons with typical energies of the order of 100 keV is therefore an essential requirement for the EC technique.

In a typical EC experiment, a crystalline sample is implanted with radioactive probe atoms of interest and the implantation damage is removed by subsequent annealing of the sample. The implanted area must not exceed a few square millimeter. Particles emitted in the decay of the implanted radioactive impurities interact with the periodic screened Coulomb potential of the surrounding crystal lattice. This interaction leads to an anisotropic flux distribution inside the crystal, often described as crystal steering effect or channeling (enhanced flux) and blocking (reduced flux) effect. Channeling and

blocking lead to anisotropic angular emission distribution within a few degrees around different crystallographic directions for particles emitted from the sample. The emission distributions are usually measured with a collimated (point-like) electron detector by rotating the sample step by step using a high precision three-axes goniometer. The emission distributions for different axial or planar directions are characteristic for the emitter atom positions within the crystalline lattice. From a quantitative analysis of the emission distributions it is therefore possible to extract the emitter atom positions with high accuracy.

For the case of decay electrons, substitutional emitters cause a strongly enhanced electron flux along atomic axes and planes, which is observed as an enhanced emission yield along these axial and planar directions. On the other hand, electrons emitted from interstitial emitter atoms exhibit a nearly isotropic flux distribution and also a nearly isotropic emission distribution. Channeling effects, i.e. an enhanced axial or planar emission yield, is therefore always an indication for emitter atoms located within certain atomic rows or planes.

A quantitative analysis of EC spectra is done by comparison with calculated emission distributions. For the case of decay electrons a description based on the dynamical theory of electron diffraction using the many-beam formalism is appropriate [1]. The motion of energetic electrons in the periodic Coulomb potential of the crystal lattice is treated as the propagation of electron wave functions by expanding the wave functions and the potentials in Fourier series and solving the appropriate Schrödinger equation.

3. Typical applications of the emission channeling technique

In the past, EC was mainly applied to study the lattice location of ion-implanted impurities, the diffusion behavior of impurities via thermally induced lattice site changes and the impurity-defect interaction. For these studies, a variety of different impurity atoms were implanted into the semiconductors Si, Ge, SiC, diamond and most of the III-V- und II-VI compound semiconductors [1,9]. Recent EC studies focused on the III-V materials GaN and AlN [10,11],

ion-implanted diamond [1,12] as well as on the investigation of transition elements like rare earths [18,13] and Cu [14] in Silicon.

The behavior of Li impurities was extensively studied using EC. In many semiconductors, Li is a fast diffusing interstitial impurity and behaves as a shallow donor. The solid solubility of Li is often very low, so that Li is preferably bound to other impurities and defects, which can lead to the electrical passivation of dopant atoms. With EC the preferred lattice sites of Li, its binding energy and the formation and stability of Li-vacancy complexes was determined using α -emitting ^8Li ($t_{1/2} = 0.84$ s) probe atoms [2].

EC was extensively applied to investigate the lattice location of implanted impurities in semiconductor materials and the annealing of implantation damage. In semiconductors like GaN, AlN and diamond the diffusion of most dopant atoms can be neglected, even at very high temperatures. Therefore, doping by ion implantation is an attractive alternative for the fabrication of GaN-, AlN- and diamond-based semiconductor devices. The behavior of implanted dopant atoms and the annealing of implantation damage in these materials is therefore of current interest [11,16].

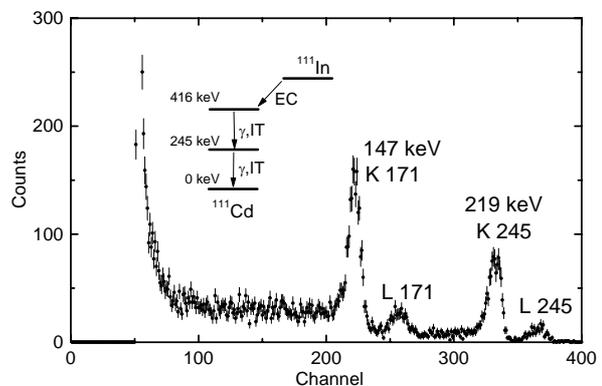


Fig. 1: Energy spectrum of conversion electrons (internal transition, IT) from the decay of ^{111}In implanted into Si, measured with a conventional silicon surface barrier (SSB) detector. The width of the 147 keV K-shell conversion electrons (from the 171 keV γ transition) and 219 keV (K 245) peaks are FWHM \approx 12-15 keV. The background is due to Compton electrons. Noise dominates below channel 60 (40 keV). The decay scheme is shown as insert.

To illustrate the information obtained from EC measurements and to introduce the ^{111}In probe atom used in the test measurements, we will briefly present

the results of EC studies for GaN and diamond following room-temperature heavy-ion implantation of ^{111}In atoms.

^{111}In ($t_{1/2} = 2.8$ d) decays to stable ^{111}Cd . In the decay, K-shell conversion electrons with energies of 147 keV and 219 keV are emitted with relative intensities per decay of 9 % and 5 %, respectively. Since the initial decay is an electron capture, characteristic Cd K- and L-X-rays are also generated. The energy spectrum of decay electrons measured with an ORTEC SSB detector is shown in fig.1.

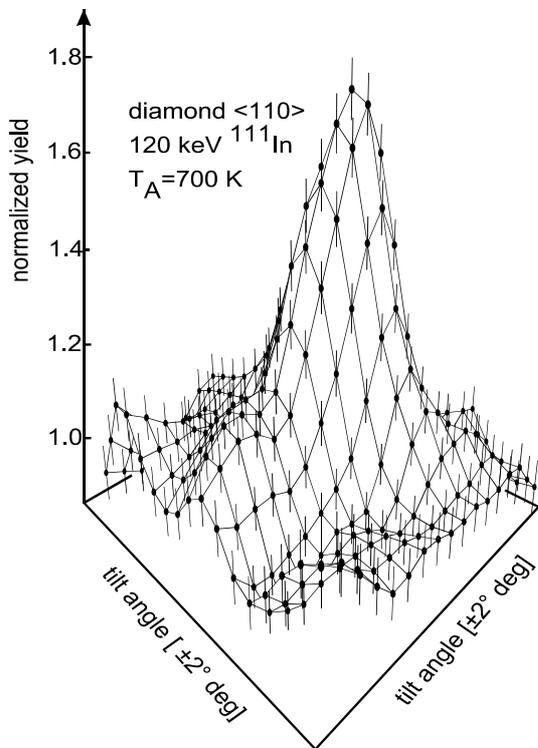


Fig. 2: Emission channeling spectrum of conversion electrons emitted from ^{111}In implanted in type IIa diamond. The spectrum was measured after implantation of $5 \cdot 10^{13} \text{ cm}^{-2} \text{ }^{111}\text{In}$ with 120 keV and annealing to 700 K. Plotted is the normalized emission yield as a function of the emission angle with respect to the <110> axis. The spectrum was obtained by tilting the sample in steps of 0.25° using a collimated SSB detector. (From ref. [1])

Channeling effects of the decay electrons, i.e. maximum emission yields along different crystallographic directions, were observed directly after room temperature implantation, indicating that

(i) the crystal lattice is not amorphized by the implantation fluence and (ii) a significant fraction of the implanted In atoms occupy substitutional lattice sites in GaN (see Fig.3, top) and diamond.

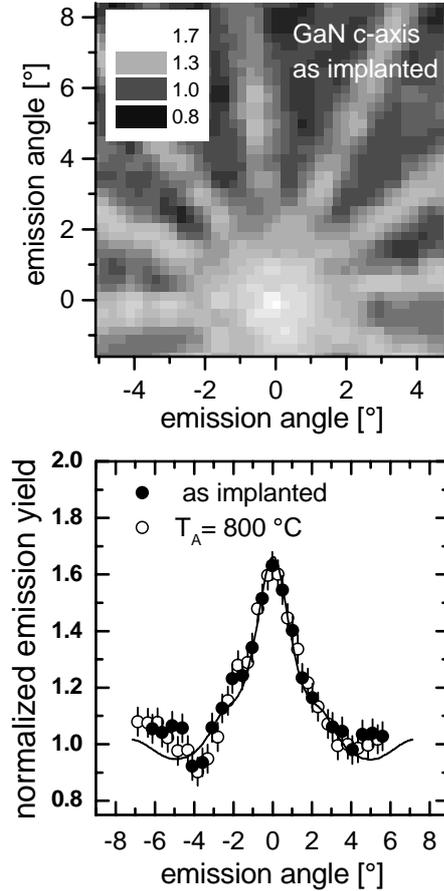


Fig. 3. Top: Emission channeling spectrum of 147 keV and 129 keV conversion electrons for the c-axis in GaN measured directly after room temperature implantation of ^{111}In . The inserted gray scale indicates the emission yield. Non-axial and non-planar orientations are referred to as random orientations and the corresponding random emission yield is usually used for normalization of the emission yield. Bottom: Random-axial-random cut through the c-axis emission distribution for the as-implanted sample and after annealing the sample to 1073 K. Solid line: many-beam simulation of the normalized emission yield assuming 90 % substitutional emitter atoms.

For diamond, isochronal annealing leads to improved channeling effects in two steps, one weak step between 300 and 600 K and another pronounced step around 1200 K. [1,12]. An emission channeling spec-

trum obtained after annealing to 700 K is shown in fig.2. The observed annealing steps can be correlated with the thermal annealing of certain implantation defects, such as interstitials and vacancies. The annealing stage at 1200 K is also observed in cathodoluminescence studies at ion implanted diamond [17]. Above 1200 K a quantitative analysis of the EC spectra reveals only about 40 % of substitutional probe atoms, indicating that simple thermal annealing is not very effective in removing implantation damage.

The situation is different for ion implantation into GaN. Following room temperature implantation into GaN, pronounced channeling effects can be observed (Fig.3), corresponding to a substitutional fraction of probe atoms above 90% [15]. Isochronal annealing to 1073 K (Fig.3 bottom) leaves the channeling effects unchanged. The high substitutional fraction of In atoms and the absence of annealing stages visible with EC are an indication that only little residual damage is present direct after implantation. Such a behavior of GaN can be explained with its high bond ionicity which prevents the formation of extended defects structures during ion implantation [16].

For other semiconductors, EC measurements reveal an annealing behavior between the two extreme cases diamond and GaN. Depending on the implanted impurity, the matrix and the local defect structure either a substitutional or an interstitial incorporation of the impurities will prevail [1].

4. Detectors for electron channeling measurements

4.1. General requirements

Conversion electrons emitted in the decay of excited nuclei with discrete energies ranging from several keV up to about 300 keV are suited for EC studies. Detection of 100-300 keV electrons requires semiconductor detectors with an active thickness (depleted zone) of about 300-400 μm and an energy resolution of about 10 keV. Channel plate detectors are not suitable as imaging detector for EC studies because of the complete lack of energy resolution. Many isotopes emit rather low energetic conversion electrons (< 20 keV) with large internal conversion

fractions exceeding 90 %. Those isotopes would be ideal for emission channeling, however their detection requires extremely low-noise semiconductor detectors with thin entrance windows and good energy resolution.

The angular width of axial and planar electron channeling effects depends on the respective crystallographic direction, the host matrix elements and the electron energy. A typical width is in the order of 1° , so that an angular resolution of at least 0.2° and a total angular range of about $\pm 5^\circ$, horizontally and vertically, is necessary. The angular resolution can be achieved for an electron detector pixel size not exceeding the typical diameter of 0.5 – 2 mm of the sample area implanted with radioactive probe atoms. The position resolution of most semiconductor detector arrays is much better, however, a pixel size smaller than about 100 μm is not necessary for electron emission channeling applications.

Most EC measurements are low count rate experiments, i.e. the impurity concentration and thus the activity of the sample is kept as low as possible. The total count rates for a 2D pixel detector may reach several kHz.

4.2. Imaging particle detectors suitable for EC

Position sensitive semiconductor detectors (PSD) based on the charge division principle have been successfully applied in EC studies for imaging of α -particles. Those detectors are large area PIN-diodes with a laterally homogeneous dopant concentration in the p- and n-type doped regions [3]. The charge readout is done via two vertically and horizontally arranged electrode pairs. An analog signal processing system using standard NIM modules is sufficient to extract the energy and the x,y-position of an incident particle. For 2×2 cm^2 PSDs (type 2L20 from Sitek, <http://www.sitek.de>) we get a position resolution of about 120 μm for 1.6 MeV α -particles. The energy resolution is at best 40 keV. Since it is a large area detector, the integral count rate should not exceed a few kHz to avoid double and multiple event pile-up. Charge division PSDs are not suited for imaging of electrons because the created charge per electron is significantly smaller compared to α -particles. This results in signals close to the noise level, a poor energy resolution and a poor position resolution.

Charge division PSDs are therefore not suitable as electron imaging detector for EC studies.

Recently so-called pad detectors have been successfully used in EC-studies by Wahl et al. [18,5]. Pad detectors are energy-resolving pixel array detectors as introduced by Kemmer et al. [19]. In EC measurements, the read-out of all pads is necessary upon a self-trigger event derived from the common back contact. In the experiments of Wahl et al. $30 \times 30 \text{ mm}^2$ detectors with 22×22 pads of size $1.3 \times 1.3 \text{ mm}^2$ were used. The active pad thickness was up to 1 mm, sufficient for detection of high energy electrons from β -decay. The self-triggered sequential readout of the pads with a total readout time of 2.5 ms limits the maximum total count rate to about 100 Hz or in average to only 0.2 Hz per pixel. Similar pad detector modules with pixel size ranging from 1 mm^2 to 50 mm^2 were developed for the PHOBOS-Spectrometer at the Relativistic Heavy Ion Collider (RHIC) in Brookhaven [20,21].

In recent years, CCD detectors were developed for imaging of soft X-rays with energies between 0.2 keV and 15 keV [6]. The main application potential for these CCD detectors lies in the areas of X-ray astronomy [22]. The requirements upon those CCD sensors are: (i) an optimum energy resolution, (ii) a high quantum efficiency for soft X-rays, (iii) a large sensitive area and (iv) a fast read-out. Criteria (i) and (ii) imply a low detector noise. All requirements are ideal presuppositions for an imaging spectroscopic detector for energetic electrons. In the following chapter we present first EC measurements with conversion electrons using an X-ray pn-CCD sensor.

5. Experiments with an X-ray CCD detector

We performed emission channeling measurements using an X-ray pn-CCD sensor developed at the MPI semiconductor laboratory in Munich. The sensor chip consists of 64 columns of 200 pixels sized $150 \times 150 \mu\text{m}^2$. The total sensor area was $1 \times 3 \text{ cm}^2$. A high quantum efficiency is ensured by a thin entrance window and a totally depleted pn-junction throughout the $300 \mu\text{m}$ wafer thickness. A high energy resolution is achieved by using highly pure FZ-Si (5 k Ωcm), integrated preamplifiers per column and cooling of the CCD sensor. The noise level at 180 K is given as

$5 e^- \text{ ENC}$. For soft X-rays from a ^{55}Fe source the energy resolution was 130 eV.

The 64 columns of the CCD sensor were read out in parallel and the signals were digitized by a 12 Bit 10 MHz ADC. The read-out time was 22.5 μs for the 64 pixels per row and 4.5 ms for the entire detector. In contrast to the pad detectors and PSD detectors discussed above, the generated charge per pixel is stored. The readout can therefore be done periodically instead of event-triggered and thus total count rates of several kHz are easily feasible.

The measurements were done with conversion electrons from radioactive ^{111}In implanted into a single-crystalline Si (100) sample. ^{111}In was implanted at room temperature at the Göttingen ion implanter IONAS with 60 keV and a fluence below $10^{13} \text{ ions/cm}^2$. Following implantation the sample was annealed to 1173 K in order to remove the implantation damage. From earlier emission channeling measurements it is known that In occupies substitutional sites in Si and we expect pronounced channeling axial effects with normalized maximum emission yield of about 2.5 along the $\langle 100 \rangle$ axis.

The measured electron energy spectrum is shown in Fig. 4. The conversion electrons with energies ranging from 147 keV up to 245 keV are clearly resolved. The energy resolution is excellent compared to the conventional detector (compare with Fig. 1). The measured FWHM for the 147 keV (K171) and 219 keV (K245) electrons are about 3 keV. This is to our knowledge the best electron energy resolution achieved with a semiconductor detector. Because of the extreme low noise level, the CCD sensor is able to detect low energy electrons as well as X-rays from the $^{111}\text{In}/^{111}\text{Cd}$ -decay.

For these measurements, the amplification of the integrated readout system was strongly reduced, in order to detect conversion electrons with energies above 200 keV without signal overflow. Therefore, the sensitivity and energy resolution of the CCD sensor is reduced, which has only an influence in the low energy regime.

For a sample-detector distance of 100 mm we finally measured the channeling effect of the conversion electrons around the $\langle 100 \rangle$ axial direction (Fig.5). Since no goniometer was available it was not possible to analyze other axial directions. For the

measurement we only used signals from the conversion electrons, i.e. the energy regime 145 – 250 keV.

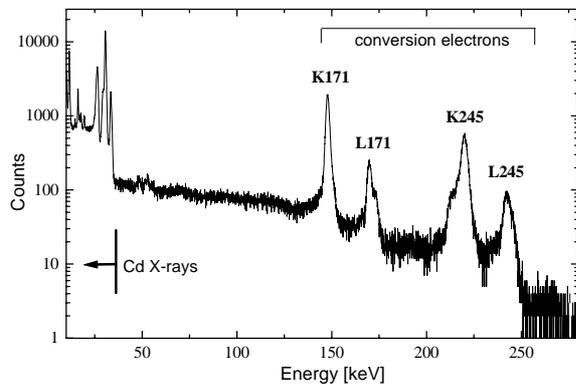


Fig.4. Energy spectrum measured with the CCD sensor, exposed to the ^{111}In implanted test sample. The K- and L-conversion electron signals appear in the high energy region of spectrum. The peaks below about 40 keV are K- and L-X-rays produced in the electron capture decay $^{111}\text{In}/^{111}\text{Cd}$. The continuous background are Compton electrons.

At the time of the measurement, the activity of the sample was rather low. Nevertheless, the axial channeling effect with the expected emission yield and also planar channeling effects for the $\{110\}$ -planes are clearly visible. The asymmetry of the spectrum along the long axis of the sensor is caused by a shadowing effect from an Al collimator mounted in front of the sample.

From the measurement with a conventional semiconductor detector we determined an activity of the sample of $2 \cdot 10^5$ Bq corresponding to about $7 \cdot 10^{10}$ ^{111}In atoms. The measurement with the CCD sensor was carried out about one week later, i.e. with a sample activity of only $3.5 \cdot 10^4$ Bq or $1.2 \cdot 10^{10}$ ^{111}In atoms. Even for such low activities emission channeling spectra of good quality can be recorded in a reasonable short time.

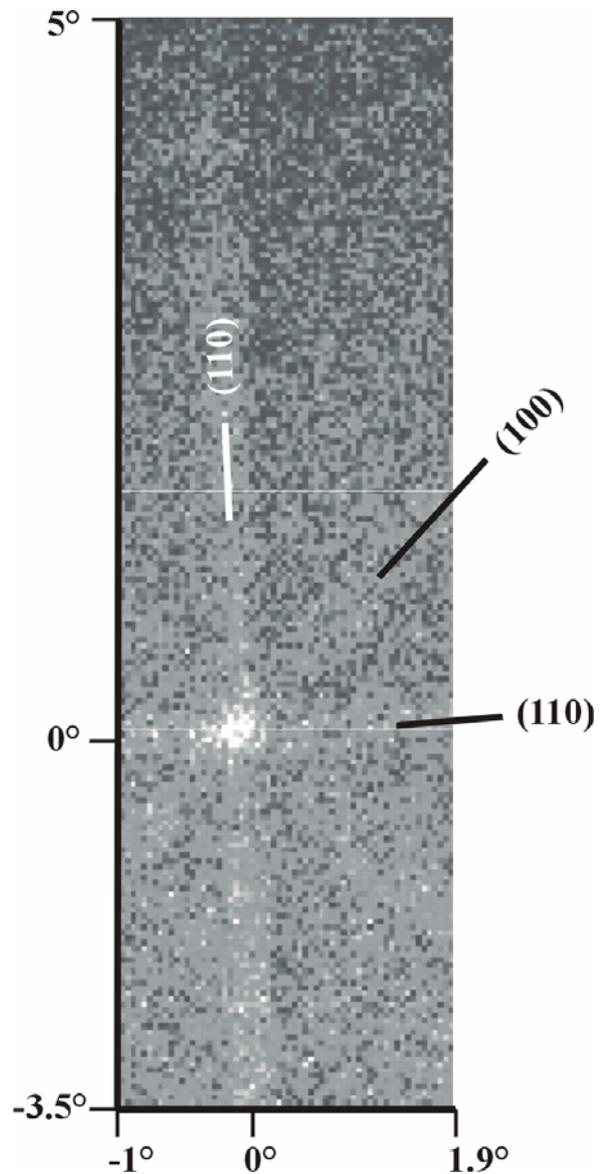


Fig.5. Emission channeling pattern of conversion electrons from the decay of ^{111}In measured along the $\langle 100 \rangle$ -axis of Si. The spectrum was measured with the 1×3 cm² pn-CCD-sensor for a sample detector distance of 100 mm and an emitting sample area collimated to 2 mm in diameter, corresponding to an angular range of 2.9° horizontally and 8.5° vertically. The axial $\langle 100 \rangle$ channeling effect and also planar channeling effects, reflecting the four-fold symmetry of the $\langle 100 \rangle$ axis, are clearly visible. Due to shadowing effects of the collimator the emission distribution has a slight asymmetry. The gray scale is given in arbitrary units. White dots represent a high count rate.

6. Conclusion

The measurements demonstrate that pn-CCD sensors are excellent imaging spectroscopic sensors for energetic electrons in the energy regime up to 300 keV. Compared to other position sensitive semiconductor detectors, the CCD sensor as an electron detector has several advantages. First, the energy resolution of 3 keV FWHM is extremely good. Second, the charge storage and the periodic fast parallel read-out allow high total count rates. Third, because of the low noise level, the CCD sensor can be used to detect low energetic electrons down to energies of about 10 keV. This opens the way to emission channeling studies using a large number of isotopes which could not be used with the current detector systems. A selection of those isotopes is given in Table 1, amongst them for example the Mössbauer isotopes ^{57}Fe und ^{119}Sn . Complementary studies using Mössbauer spectroscopy and emission channeling may then become possible.

Table 1

Compilation of radioactive isotopes emitting low energetic conversion electrons. The fraction of internal conversion processes per decay is given in column 4. The kinetic energy of the emitted electrons is listed in column 5.

1	2	3	4	5
Isotope	Parent	half life	fraction [%]	energy [keV]
57-Fe	57-Co	271.8 d	80	7.3 –13.7
58-Co	58m-Co	9.15 h	99.67	17.2 –24.1
73-Ge	73-As	80.3 d	> 90	2.2-52.2
103-Rh	103-Pd	16.99 d	99.3	16.5-37
103-Rh	103-Ru	39.26 d	99	16.5-37
119-Sn	119-Sb	38.2 h	83.9	19.7
125-Te	125-I	59.4 d	93.3	3.7-30.9
129-I	129-Te	69.6 m	83.7	23.9
149-Sm	149-Eu	93.1 d	97.68	15
151-Eu	151-Sm	90 y	85	14
160-Ho	160-Er	28.6 h	99.9	4.4-51
161-Dy	161-Tb	6.88 d	> 60	3-66
166-Tm	166-Yb	56.7 h	85	22.9 –72.7
167-Er	167-Tm	9.25 d	> 25	47.7-198.7
169-Tm	169-Yb	32 d	40	3.6 - 53.4
171-Yb	171-Lu	8.24 d	96.3	9.4
181-Ta	181-Hf	42.4 d	50	65.5 -122
183-W	183-Re	70 d	> 25	35-150.5
189-Os	189m-Os	5.7 h	100	18.4
195-Au	195-Pt	186 d	80	84.3

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