

Charged particle detectors made of single-crystal diamond

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Very first test results obtained from an intrinsic single crystal CVD diamond detector are described and compared to those measured with polycrystalline CVD-diamond and HPHT Ib diamond sensors. The data are discussed in the context of the requirements of intermediate and high energy heavy-ion physics experiments. It is shown how the crystal structure and the chemical purity of diamond material affect the detector properties as well as vice versa how the detector response to impinging charged particles can be used to study the homogeneity of the mass density and the electrical properties of diamond.

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1 Introduction

Diamond Detectors (DD) in Heavy-Ion (HI) and high-energy physics experiments are usually being considered for charged particle detection whenever the experimental requirements on the radiation hardness and the speed of sensors exceed the abilities of the classical detector devices. Although silicon is still the standard material for solid state detectors, the excellent timing and good tracking results of the Poly Crystalline CVD-Diamond Detectors (PC-DD) [1, 2] have finally got more attention of the potential users. However, the inhomogeneity of the polycrystalline material leads to serious disadvantages limiting the implementation of such devices in a broader field of detector applications. The incomplete and position dependent charge collection affects both, the energy and the time resolution of the detectors, introducing additional background due to secondary reactions in the ‘dead layer’ of the samples. Spatial resolution and detection efficiency are disturbed as well. For instance, if traversing grain boundary regions, focused beams can pass through the counters completely undetected.

Striving for radiation-hard HI DD of superior performance in both, time and energy resolution, we suggest a solution to the inhomogeneity problem through the use of single crystal diamond as detector material. We have assumed that HPHT type IIa diamond as well as HPHT type Ib would have the properties needed. In a joint research project with the Schonland Research Institute for Nuclear Science in South Africa [3] we investigated Ib type DD with nitrogen impurities of the order of hundreds of ppm but hitherto unavailable perfect crystal structure [4] for this purpose. However, recent results reported from detectors made of free standing undoped Single Crystal CVD Diamond (SC-DD) grown on HPHT-Ib substrates are more promising [5, 6].

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In this report, first characterisation data obtained from such a SC-DD sample of an area of $3.3 \times 4.9 \text{ mm}^2$ and a thickness of $440 \text{ }\mu\text{m}$ [7] are compared to those measured with polycrystalline diamond sensors. Additional results from HPHT-Ib-DD should complete the view on artificial DD we have at present. It is the aim to show that R & D on detector and front end electronics is a useful method to characterize particularly the electrical properties of diamond material combined with a realistic technological task.

2 Characterisation of a SC-DD sample using detector test results

2.1 I–V characteristics and plateau measurements

A charged particle DD is a diamond sample metallized with sandwich electrodes connected via printed circuit board and coaxial connectors to a power supply. When charged particles pass through e–h pairs are created, which become separated and move towards the electrodes guided by the external electric field E_D . The collected charge Q_C obtained from every single particle is proportional to the average drift distance of electrons and holes, the so called Charge Collection Distance (CCD) given by Eq. (1):

$$\text{CCD}^{e,h} = v^{e,h}(E_D) \tau^{e,h} = (\mu^{e,h} E_D) \tau^{e,h} \quad (1)$$

where $v^{e,h}$ is the velocity, $\mu^{e,h}$ the mobility, $\tau^{e,h}$ the life time of the charge carriers and E_D the external electric field.

A $\text{CCD}^{e,h} \leq d_D$ is measured according to Eq. (2):

$$\text{CCD} = \frac{Q_C}{Q_G} d_D = \text{CCE} d_D \quad (2)$$

where Q_C is the collected charge, Q_G the primary ionisation charge produced by each particle and d_D the detector thickness. The ratio Q_C/Q_G is the measured Charge Collection Efficiency (CCE) of a detector.

IV characteristics as well as detector Plateau measurements must be performed in order to define the bias range of operation of a detector. This is the range of highest possible CCE and speed, and of low dark current. According to Eqs. (1) and (2) the electric field needed to achieve maximum CCE is the same as required to saturate the carrier velocities. In the following, hysteresis measurements of the voltage dependent leakage current and of the CCE of the SC-DD are discussed. The data are compared to those obtained from an ‘as grown’ PC-DD of a thickness of $388 \text{ }\mu\text{m}$ and an area of 1 cm^2 . A Keithley 6517 electrometer is used for the IV measurements and charge sensitive electronics for the measurement of the CCE. The electrodes of the SC-DD are made by evaporation of Ti–Pt–Au (100–100–200 nm) whereas those of the PC-DD by evaporation of Cr–Au (50–200 nm). In both cases no annealing is performed after metallization.

In Fig. 1 data obtained from virgin samples are shown. The measured dark current is normalized to the corresponding active detector volume and is plotted in Fig. 1a) against the external electric field E_D . An unexpected low break-down field of $E_{\text{break}}^{\text{SC}} = \pm 0.15 \text{ V}/\mu\text{m}$ has been observed for the SC-DD, whereas the PC sample show an asymmetric but wide bias range of $-0.9 \leq E_{\text{break}}^{\text{PC}} [\text{V}/\mu\text{m}] \leq 2$.

Figure 1b) shows the plateau characteristics of the SC-DD compared to the Plateau of a polished detector-grade PC-DD of a thickness of $500 \text{ }\mu\text{m}$. β particles from a ^{90}Sr source are used for these measurements. ^{90}Sr electrons show a continuous energy distribution with an E_{max} of 2.4 MeV . Triggered by a plastic scintillator detector only electrons of an energy $>1.5 \text{ MeV}$ (assumed to be Minimum Ionizing Particles (MIP)) are accepted. A remarkable low saturation field of $E_{\text{sat}}^{\text{SC}} \approx 0.05 \text{ V}/\mu\text{m}$ and a completely saturated Plateau have been recorded, never obtained from PC-DD.

Although the unexpected low measured volume resistivity indicates surface problems of the SC device, it is unlikely to assume that the symmetry of the CCE data with respect to the bias polarity, the lack of a hysteretic behaviour and especially the prompt and complete saturation of the plateau are obtained also due to such device problems. To our understanding, the data indicate at least a homogeneous distribution of electron and hole traps, if not in addition an extreme low concentration of it inside the bulk material.

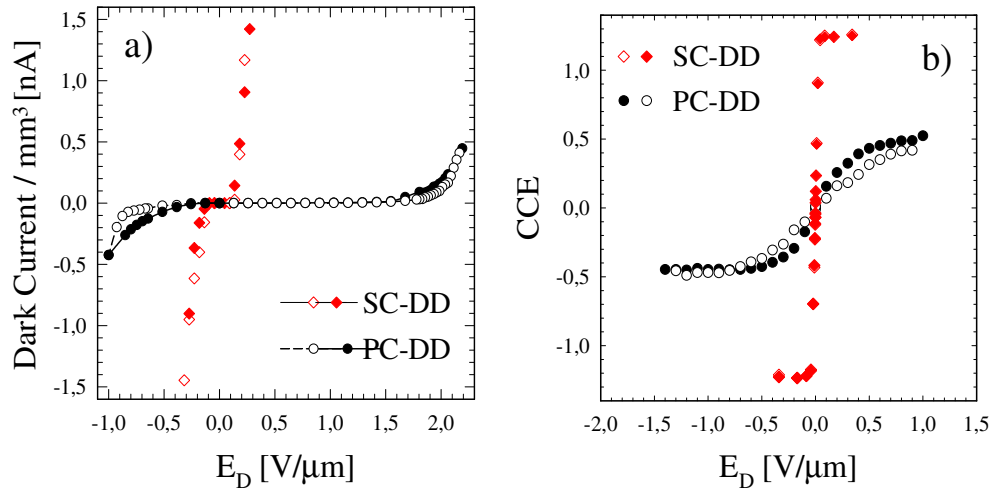


Fig. 1 Hysteresis measurements of the dark current per mm³ (a) and of the CCE (b) as functions of E_D . A symmetric behaviour of the data and a fully saturated CCE plateau have been obtained in the SC case. The unexpected low $E_{\text{break}} \ll 0.5 \text{ V}/\mu\text{m}$ may be caused from surface conductivity. However, a CCE > 1 is not conclusive yet (see text).

Nevertheless, the measured CCE which is higher than 100% at saturation opens questions. Two explanations are possible: reinjecting contacts (see also sect. 2.3) or calibration problems. According to various reports [2, 5] a value of $36 \text{ e}^-/\mu\text{m}$ has been used for calibration. This is assumed to be the charge generated by a MIP in diamond. The data may indicate that this value is not valid or that electrons of lower energy which produce more excess charge have been triggered by the scintillator detector. However, the unexpected results described in the following allow for other explanations as well.

2.2 Thermally Stimulated Current Measurements (TSC)

TSC measurements are performed after irradiation of the samples with a ⁹⁰Sr source as long as 30 min in both cases. This procedure leads to a filling of a large amount of existing traps in the case of polycrystalline diamond improving the CCE of the detectors by a factor of 2 [8]. Equal CCD has been measured before and after irradiation for the SC-DD.

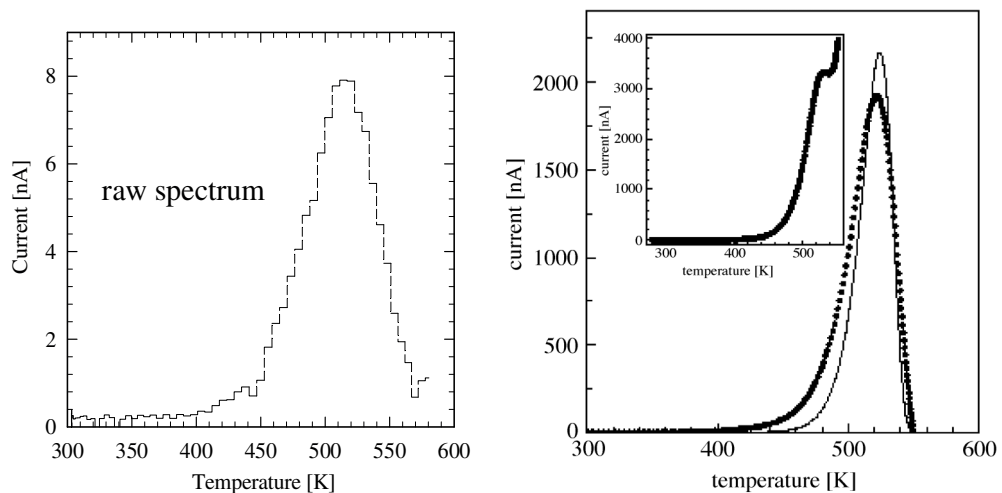


Fig. 2 Thermally stimulated current measured with a SC-DD (left) and a PC-DD (right). Only a qualitative comparison is attended. The TSC peaks appear on the same temperature.

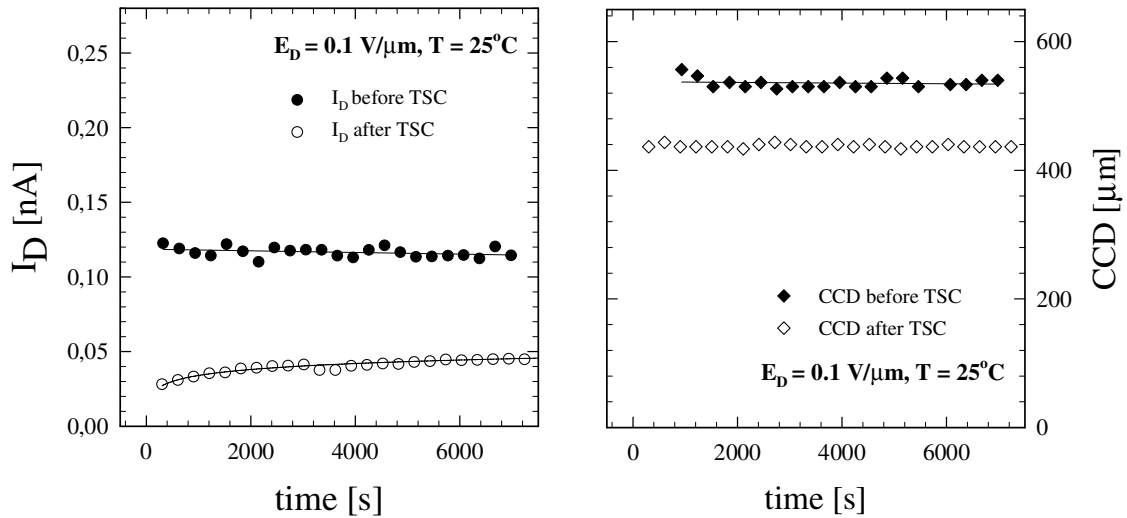


Fig. 3 Stability of dark current and collection distance of the SC-DD before and after TSC measurements. The high CCD $> d_D$ measured before heating decreases to its expected value of 100%, i.e. CCD = 440 $\mu\text{m} = d_D$.

The thermally stimulated current has been measured by heating the sample with a gradient of 1 $^{\circ}\text{C}/\text{s}$ up to 600 K. The analysis of the SC-DD spectrum is not performed yet. Figure 2 shows on the left the raw spectra obtained from this sample and on the right well analyzed data from a detector grade PC-DD of a thickness of 500 μm [9]. The solid line of the latest is a fit to the experimental data based on the theoretical shape. In the insert the raw data of this measurement are depicted. The intention at present is only to show that the shapes of both raw spectra are similar and that a TSC peak appears in both cases at same temperature. Nevertheless, the data allow to assume that only little dark current is stimulated in SC CVD diamond up to this temperature.

2.3 Stability of dark current and charge collection distance

Stability tests have been performed before and after heating of the SC-DD for TSC measurements up to 600 K (Sect. 2.2). Figure 3 shows the leakage current I_D (left) and the CCD (right) obtained over two hours at constant room temperature of 25 $^{\circ}\text{C}$ and constant bias of $U_D = +50$ V on the detector. The detector parameters are stable within the time interval measured. The fits to the data are linear regressions except in the case I_D after heating, which shows a weak logarithmic rise to a flat maximum.

The dark current decreases after heating by a factor of 2.6 and the CCD by a factor of 1.2 to the expected value of 440 μm , which corresponds exactly to the detector thickness. It is not clear whether the post annealing due to the TSC measurements influences more shallow traps existing in the diamond bulk or the interface to the metallization. By tuning the bias above 70 V however, the same unexpected CCE characteristics (with CCD $> d_D$) is obtained as before heating.

All measurements must be performed again with the remetalized sample after a post treatment of the device surfaces as well as an improved cleaning procedure before metallization.

2.4 Single particle time resolution: A measure for the charge carriers lifetime and mobility

Heavy ions produce such a high amount of e–h pairs in solid-state detectors that even incomplete collected charge signals are strong and above electronic threshold. Therefore, high-frequency broadband electronics can be used for timing devices maintaining such the original fast diamond response [10, 11].

Similar to the ToF (Time-of-Flight) experiments developed by W. E. Spear [6, 12] with short-pulsed UV-lasers, detector signals obtained from low energy heavy particles stopped in a few micrometers dia-

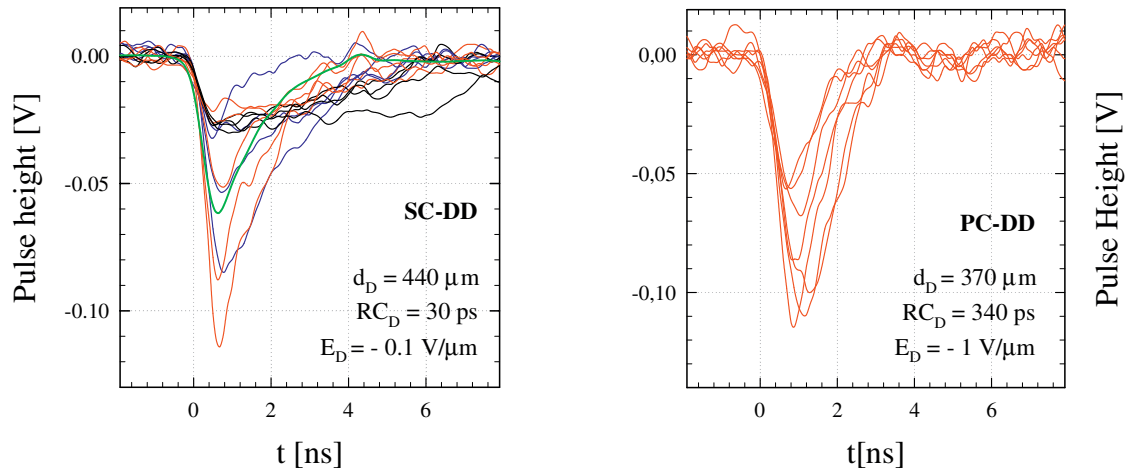


Fig. 4 Single-particle pulses from a ^{241}Am source with $E_\alpha = 5.48$ MeV as recorded with the SC-DD (left plot) and a PC-DD (right plot) using high-frequency broadband amplifiers and a 1.5 GHz DSO. The pulses do not represent the whole α -distribution and the correct S/N ratio. They are selected in order to illustrate typical shapes.

mond material can be used for the measurement of mobility and lifetime of the charge carriers. The generated e–h pair cloud is concentrated at Bragg maximum near the surface of the detector. Thus, the shape of recorded single particle pulses characterizes the transmission of the free carriers not immediately ‘absorbed’ by the electrode next to the generation point. The ultra fast rise time of the pulses defines the time of separation and the START of the charge movement, whereas the time at which the amplitude of the signal is decreased to its $1/e$ -value defines the transition time t_T , i.e. the STOP of the movement due to the absorption of the cloud either in traps (lifetime $\tau < t_T$) or from the opposite electrode (lifetime $\tau \geq t_T$). If a diamond signal is processed with front end electronics of infinite bandwidth, the mobility can be estimated from Eqs. (1) and (2) considering τ as t_T , i.e. the full width of the $1/e$ decrease of the amplitude.

Alpha particles emitted from an ^{241}Am source ($E_\alpha = 5.48$ MeV) are stopped in 12 μm diamond material. Tuning the velocity of the ions shorter ranges can be achieved with HI beams. Nevertheless, since $d_D \gg \alpha$ -range, α tests performed in the laboratory are useful as well. A Digital Storage Oscilloscope (DSO) of 1.5 GHz band width and of a sampling rate of 10 GS/s is used for the measurements. Negative bias fixing the holes is applied to the electrode impinged by the particles. The electron- α -signals obtained from the SC sample and from an ‘as grown’ PC-DD of a thickness of 370 μm and of a CCD of 92.5 μm are shown in Fig. 4. The pulses are processed with low-noise Diamond Broadband Amplifiers (DBA) of a band width of 2.3 GHz at -3 dB [11].

Unexpected different shapes of α signals have been obtained from the SC-DD supposed to be homogeneous. Two signal parameters should carefully be analyzed for a deeper understanding of this preliminary result: the area of each signal that corresponds to the collected charge from each α particle and the width of the signals illustrating the transition time t_T through the detector, given thus the mobility of the charge carriers. Taking into account the CCD measured with charge sensitive electronics and the ^{90}Sr source (Sect. 2.1) the electron mobility can be estimated using Eq. (1).

The contribution of the measurement system to the signal shape parameters is an RC_{meas} of 450 ps. The RC_D constants of the detectors can be calculated considering the detector capacitances ($C_D^{\text{SC}} = 0.6$ pF, $C_D^{\text{PC}} = 6.8$ pF) and the 50 Ω impedance of the amplifier.

In order to test the suitability of a DD for different applications, distributions of the single particle pulse shape parameters are studied: A fast and uniform rise time can be exploited for a precise definition of time as required for start detectors defining the time zero of a reaction of interest. For the counting of high particle rates on the other hand, a short ‘dead time’, i.e. a narrow total width of the signals is needed.

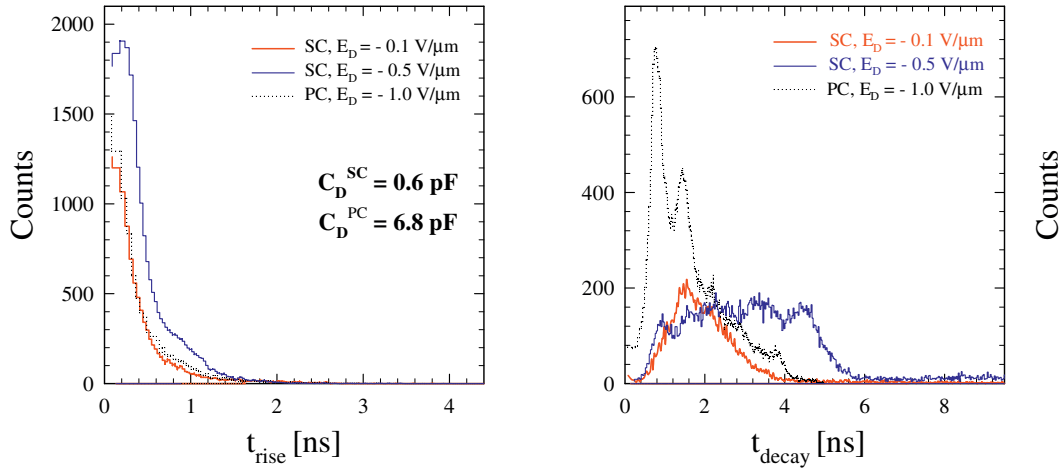


Fig. 5 Rise and decay time distributions of the SC-DD at different bias compared to the shape parameters of signals obtained from an ‘as grown’ PC-DD at $E_D^{PC} = -1$ V/ μm . The spectra are corrected taken into account the contributions of the electronics and the decay constants RC_D of the detectors.

Finally the uniformity of the broad band signals area gives a hint to the homogeneity of the materials mass density and electrical properties and thus to the energy resolution of a detector.

Figure 5 shows the measured rise (left) and decay time (right) distributions after correction with respect to the dominating contribution of RC_D and of the electronics. In the case of the SC-DD, spectra obtained at two different electric fields are presented. Note that these measurements have been performed at room temperature after heating for TSC tests. The decay time distribution of the SC-DD at an $E_D = -0.1$ V/ μm indicate charge carriers with a mean life time of about 2 ns corresponding to an enormous high electron mobility of $\mu_e^{SC} = 2.2 \times 10^4$ cm²/Vs. About 4500 cm²/Vs have been measured [6] at very low bias. The processes induced by a field higher than E_{sat} are not understood yet. Field emission in the diamond bulk or in the interface to the contacts cannot be excluded. Note that due to the AC coupled broad band amplifier used an increased leakage current in the order of nanoamps does not affect the signal performance. On the other hand, the bandwidth of the amplifier optimized for the PC-D material is not able to maintain broader signals. A simulation of the DBAII amplifier used shows that an input signal of a decay time of 100 ns appears as having a decay time of only 20 ns. In other words the DBAII amplifier has to be modified and the measurements must be repeated in order to estimate the correct mobilities. For the PC-DD, two pronounced lifetimes have been obtained. However, since the average CCD can be considered a $\mu_e^{PC} = 617$ cm²/Vs is estimated in this case.

The data demonstrate the limits of the electronics but nevertheless the excellent suitability of both types of CVD-DD for HI timing applications. The ultra fast rise times cannot be defined reliably. Due to a mean value of 1 ns and a width of 0.8 ns measured for the decay time of the PC sample, this DD is able to count rates from one single-particle up to 10^9 particles per second (pps). Lower rate capability is expected from SC-DD. The width and the structure of those distributions are the parameters characterizing the homogeneity of the electrical properties of the diamond material. They contain information concerning the homogeneity of the dielectric constant ϵ_r as well as the existence of discrete mobility values inside the bulk material. The latest is impressively demonstrated in the case of the PC-DD and just indicated for the SC-DD. The data analysis and the modification of the amplifiers are under progress.

2.5 Energy-Loss Resolution: A result of CCE and the homogeneity of the crystal structure

The most important issue in accelerator physics research is the identification of the reaction products after the collision of the projectiles with the target atoms. The multiplicity of one event in HI experiments can vary from 2 (elastic reaction) up to >2000 created particles. The event constituents are heavy

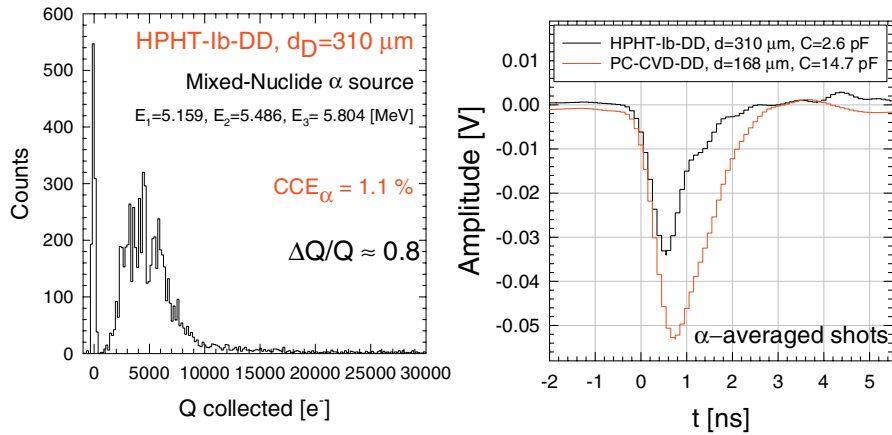


Fig. 6 (Left) Alpha spectrum of a mixed nuclide source measured with a HPHT-Ib sample of 310 μm thickness using spectroscopy electronics. The CCE amounts to only 1%. The low S/N ratio is probably the reason for a similar energy resolution as obtained for PC-DD [13]. (Right) Average broadband signals measured with DBA amplifiers.

particles (e.g. $^{238}\text{U}_{92}$, $^{208}\text{Pb}_{82}$, $^{197}\text{Au}_{79}$) emitted from projectile- and target nuclei down to singly charged protons, electrons, pions and kaons as well as high-energy photons and neutrons. The ‘nature’ of a particle is identified by its nuclear charge Z , the mass number A , the atomic charge state q reducing Z to an effective value Z_{eff} , and the behaviour of the particle in electrostatic or magnetic fields.

The specific energy loss dE/dx is a sensitive observable for particle identification as far as all charge generated and collected from each particle corresponds to the characteristic total ΔE signal which is expected according to the simplified Bethe–Bloch relation given in Eq. (3):

$$-\frac{dE}{dx} \propto \frac{Z_{\text{eff}}^2}{\beta^2} \left[\ln \frac{2m_e c^2 \beta^2}{I(1-\beta^2)} - \beta^2 \right] \quad \text{with} \quad \beta = \frac{v}{c} \quad (3)$$

where Z_{eff} and v describes the effective charge and the velocity of the particle, m_e the electron mass and I the mean excitation energy of the target atoms.

The measured pulse height distributions are calibrated in electrons by taken into account the w value of 13.4 eV which is assumed to be the energy needed to create an e–h pair in diamond [2, 5]. They are nothing else than spectra of the charge collected from each particle according to the corresponding local collection distance. Since the expected ΔE signal of a HI is well defined (Eq. (3)) the distributions measured with high resolution silicon detectors at intermediate energy measurements are narrow Gaussians of a width given mainly by the Signal-to-Noise ratio (S/N). In order to distinguish two neighbour elements of the periodic system e.g. Uranium ($Z = 92$) and Protactinium ($Z = 91$) the pulse-height resolution of a $\sigma/DE \leq 0.003$ is required, where σ is the width of the measured distributions and DE the difference of the energy losses of the two elements. Due to the same $Z = 1$ MIPs are not designable via ΔE .

Constant primary charge generation requires pure detector material of homogeneous mass density, whereas a complete charge collection can only be achieved in the absence of traps and by a uniform shape of the original detector signals (Sect. 2.4).

In order to demonstrate the influence of the diamonds purity and structure to the energy resolution the distribution obtained from an HPHT-Ib diamond sample of best crystal quality using a mixed nuclide α source (^{239}Pu , ^{241}Am , ^{246}Cm) is presented in Fig. 6. The energy intervals between the three lines are 327 keV and 318 keV, respectively. Whereas silicon detectors can easily identify the different α -energies no discrete lines are visible in this spectrum. The influence of the nitrogen contain dominates the S/N ratio.

3 Conclusions and outlook

Preliminary test results obtained with a single crystal CVD diamond sample confirm the high CCE of 100% and indicate the high electron mobility reported from other authors [5, 6]. The saturation of the charge carrier velocities occur at a very low electric field of only $E_{\text{sat}}^{\text{SC}} = \pm 0.05 \text{ V}/\mu\text{m}$. Processes induced by higher fields ($E_{\text{D}} > E_{\text{sat}}^{\text{SC}}$) must be investigated in more detail as well as the influence of surface finishing and metallization technique. However, no polarisation and no ‘priming’ of the sample have been observed.

The electronics available at present limits the detector results. In order to exploit the enormous electron mobilities for particle physics experiments, novel electronic devices (based on diamond?) must be developed. Nevertheless, this feature in conjunction with the high CCE of SC-DD provides a unique tool for charged particle timing applications. Due to a much lower S/N ratio of PC-DD, fast timing with minimum ionizing particles using DD was prevented. The reported preliminary data are promising that SC-DD meet the requirements of time-zero detectors for hadron as well as for HI physics experiments.

The broad width of the measured decay time distributions indicate inhomogeneity of the electronic parameters of both materials, which can affect the energy resolution of the detectors. Reliable measurements of the energy resolution of SC-DD are not available at present. DD made of highest quality HPHT-Ib diamond show that a perfect crystal structure does not mask the disadvantage of other defects. However, it must be clarified why a such pure diamond material shows such a low break-down field (surface conductivity?).

A basic assumption to these detector developments is the radiation hardness of SC-DD able to meet the requirements of experiments at the next generation high-luminosity accelerator facilities. A fluence of roughly 10^{15} hadrons/cm² without degradation of the detector performance has been confirmed [5] for PC-DD. Irradiations with different charged particles including neutrons and photons up to a fluence of 10^{16} particles/cm² are planned for the next three years. However, the implementation of such devices in real particle physics experiments will be limited to only a few applications if the size of synthetic SC diamond does not exceed the areas of a few square millimetres available at present.

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