Charge carrier properties of single-crystal CVD diamond up to 473 K

Benjamin Kraus, a,b, Patrick Steinegger, a,∗, Nikolay V. Aksenov, c, Rugard Dressler, a, Robert Eichler, a,b, Erich Griesmayer, d,e,f, Dominik Herrmann, a, Andreas Türler, b, Christina Weiss, d,f

a Laboratory of Radiochemistry, Paul Scherrer Institut, Forschungstrasse 111, 5232 Villigen PSI, Switzerland
b Department für Chemie und Biochemie, Universität Bern, Freiestrasse 13, 3012 Bern, Switzerland
c Flerov Laboratory of Nuclear Reactions, Joint Institute for Nuclear Research, Joliot-Curie 6, 141980 Dubna, Russian Federation
d CVI/DEC Instrumentation GmbH, Schottengasse 3A/1,1410 Vienna, Austria
e Faculty of Electrical Engineering and Information Technology, Vienna University of Technology, Gusshausstrasse 25-29, 1040 Vienna, Austria
f Faculty of Physics, Vienna University of Technology, Stardianalle 2, 1020 Vienna, Austria

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ABSTRACT

The drift behavior of charge carriers, generated by α-particles of a reference 241Am-source, in electronic grade, single crystal chemical vapor deposition (scCVD) diamond was investigated by the transient current technique (TCT) from room temperature up to ≈ 473 K. Furthermore, the α-spectroscopic behavior was analyzed in terms of charge collection and spectroscopic resolution for the same temperature range. All conducted measurements revealed complete charge collection up to the maximum temperature. The electron–hole-pair creation energies were derived from the TCT as well as from the spectroscopic measurements. The herein presented results imply that high temperature α-spectroscopy with diamond-based semiconductor solid state detectors, using presently available scCVD sensor substrates, is feasible at least up to 473 K. Only at the highest applied temperature, the conducted TCT measurements showed distorted signal traces, indicating a uniform positive space charge build-up.

1. Introduction

Diamond has been in the focus of experimentalists working in harsh environments. Its large indirect band gap of 5.47 eV [1] at room temperature in combination with the associated, favorable charge carrier properties in the bulk make diamond a superior material for detectors used in intense radiation fields or at high temperatures. Thus, diamond has been considered as detector material for the employment under such demanding environments, e.g., in high luminosity experiments at CERN in the form of TCT-based beam loss monitors at cryogenic and ambient temperatures [2], in the International Thermonuclear Experimental Reactor, ITER, for spectroscopic measurements at high temperatures [3] or in nuclear power reactors in general [4]. A further field of application is its use as detector material for α- and spontaneous fission spectroscopy in transactinide research, where newly discovered elements are characterized at the one-atom-at-a-time level using online gas-adsorption chromatography experiments [5,6]. While the performance of diamond-based semiconductor solid state detectors is rather well studied for low temperatures up to and a little above 300 K [7], studies on the charge carrier properties at higher temperatures remain scarce [8,9].

However, the high-temperature (HT), α-spectroscopic performance of diamond-based semiconductor solid state detectors has been studied since the late 1970s, initially with sensors made of natural diamond [10] and later on with synthetic material of improving quality [3, 11–14]. To the best of our knowledge, the currently highest temperatures for α-spectroscopic measurements were reported in [15–17].

Herein we report on the continuation of our previous experiments [14], thus providing further insights on the HT behavior of diamond-based particle detectors up to 473 K. For the investigation of the charge carrier properties of both, electrons e− and holes h+, we are using the transient current technique (TCT, see [19] or [20] for more details) at a nearly uniform electric field of ≈ 1 V/μm throughout all conducted measurements. Employing fast electronics with a bandwidth of 2 GHz allows for recording the very short rising and falling times of the signal edges. Until today, such measurements have been primarily performed from room temperature [20,21] down to cryogenic temperatures of 2 K [7]. Results from α-particle-based TCT measurements at temperatures well above 300 K are missing and therefore part
of this study (for measurements using optical excitation, see [8,9]). From the gathered data physically meaningful measures are extracted, thus characterizing the charge carrier behavior in diamond within the mentioned temperature range. In addition to the TCT measurements, the general α-spectroscopic performance is addressed for the same temperature range. Thereto, a charge sensitive preamplifier is employed in order to characterize the energy response using standard pulse height resolution as well as to study the resulting energy resolution in the obtained α-spectra.

2. Experimental setup

The diamond sensor used in the course of the herein presented experiments was manufactured by CIVIDEC Instrumentation GmbH from a bare electronic grade, single crystal chemical vapor deposition (abbreviated in the following with scCVD, i.e., referring to the electronic grade type) diamond substrate, provided by Element Six Ltd. This scCVD diamond sensor with outer dimensions of 4.61(1) × 4.61(1) × 0.47(1) mm³ (note the concise uncertainty notation, where 0.47(1) mm corresponds to 0.47 mm ± 0.01 mm) was set up in a parallel plate geometry with 3 × 3 mm² ohmic contacts on both sides, each consisting of 120 nm Ti, followed by 100 nm Pt, and a final outermost layer of 250 nm Au. The diamond sensor was placed in a polyimide-based, three-layered printed circuit board (PCB) structure, which ensured a temperature stability up to ≈ 500 K (see Fig. 1). The electrical contacting was established on the back side with an Au-plated (1 μm) Cu/Be-spring (98% Cu and 2% Be), thereby pressing the scCVD diamond sensor onto the grounded pad in the front (see Fig. 1). A Kapton-insulated coaxial cable was mechanically fixed on the signal and ground pads of the PCB structure and connected to the outside of the vacuum housing via a LEMO type 00 plug and vacuum feed-through. These electrical connections had a length of ≈ 90 mm with a total capacitance of ≈ 20 pF (including sensor, contacts, cable, and stray capacities).

The top part of the detector structure itself was enclosed in a two-part heat spreader made of copper (see Fig. 1). Both parts could be resistively heated, each with a cylindrical 250 W heating cartridge (MaxiWatt), enabling maximum temperatures of ≈ 1000 K. The temperature was measured in close proximity to the scCVD diamond sensor by means of a type K thermocouple, pushed through an insert of the PCB structure (see Fig. 1). As the sensor unit was firmly pressed onto the underlying PCB and due to the excellent heat conductivity of diamond itself, the measured temperature in the PCB corresponded to the temperature of the scCVD diamond sensor within ±3 K. The temperature uncertainty was verified prior to the actual experiments with a cross-comparison between the main thermocouple and another one mounted in the position of the sensor.

The detector assembly with its heating jacket was mounted inside a vacuum chamber, which could be pumped down to minimum pressures of at least 10⁻⁴ mbar. A reference ²⁴¹Am-source with an activity of ≈ 15 kBq and a main α-line at $E_{\alpha_1} = 5485.56(12)$ keV [22] was fixed on a thermally insulated mount at a distance of 45 mm from the front face of the sensor. The α-particles reached the scCVD diamond sensor through small openings in the Cu heat spreader and the front side of the PCB structure. The energy loss in the metal electrode and the stopping range of the α-particles in diamond were estimated using the SRIM 2013 software package [23]. Under the assumption of no intrinsic energy loss in the source itself and due to residual gas, the α-particles entered the active volume of the scCVD diamond sensor at an average energy of $E_\alpha = 5304(14)$ keV after passing through the three-layered metalization. With this sensor setup, the α-particles were stopped in the bulk at a mean penetration depth of 13.4(2) μm, i.e., close to the entrance electrode and at a short distance in comparison to the overall thickness of the sensor.

All herein presented measurements were performed with amplifiers from CIVIDEC Instrumentation GmbH. For the TCT measurements, the C2-HV broadband current amplifier with nominal parameters of 2 GHz analog bandwidth, a gain of 40 dB, and an input impedance of 50 Ω at an equivalent noise charge (ENC) noise of 0.4 μA ms was used. Meanwhile, the spectroscopic behavior was studied employing the unipolar charge-sensitive Cs spectroscopic amplifier with a Gaussian shaping time of 1 μs full width at half-maximum (FWHM) and a gain of 17.8 mV/fC at an ENC noise of 300 electrons from the amplifier plus an additional 30 electrons/pF accounting for the electrical connection. The scCVD diamond sensor was biased at ±500 V (i.e., ±1.06 V/μm) using a Keithley 2410 SourceMeter with the polarity set depending on the aimed at charge carrier drift through the bulk of the sensor. Finally, the data was recorded with a LeCroy WaveRunner 640Zi oscilloscope with 8.5–11-bit vertical resolution and a signal-to-noise ratio (SNR) of 55 dB. For each temperature step, 100 waveforms of α-particle-induced signal traces were stored during the TCT measurements (10 GS/s) opposed to 1000 waveforms in case of the spectroscopic measurements (250 MS/s).

3. Data analysis

The waveforms recorded during the TCT measurements with the current-sensitive amplifiers were analyzed with an approach similar to the one outlined by Pernegger et al. [20] and used in [7]. The 100 individual voltage traces $U_m(t)$ for each temperature step were converted according to Eq. (1) with the electronically calibrated values for the gain $R_{\text{amp}} = 155.9$ and the input impedance $R_{in} = 52$ Ω. The resulting current pulse $I_m(t)$ is given as

$$I_m(t) = \frac{U_{m}(t)}{R_{in} \cdot R_{\text{amp}}}$$  \hspace{1cm} (1)

The average drift time $t_d$ as well as the collected charge $A_i$ for each temperature step were extracted as the FWHM and the integrated area, respectively, from individual fits to all 100 pulses (possible outliers were excluded programatically using the generalized extreme Stuendtized deviate test with $a = 0.05$ [24]). Fitting all traces individually allowed for the evaluation of the variations of the FWHM as well as of the integrated area at a given temperature. As the charge carriers are
creased along the track of penetrating α-particles close to the entrance electrode, the drift velocity $v_{dc}$ can be evaluated by dividing the sensor thickness $d = 470(10)$ μm by the drift time $t_c$:

$$v_{dc} = \frac{d}{t_c} \quad (2)$$

For the spectroscopic part of this study, a charge-sensitive amplifier was used (see Section 2) to record 1000 pulses at each detector temperature (outliers excluded according to [24] and using $\alpha = 0.05$). These were then analyzed using standard pulse height analysis for the determination of the individual amplitudes $U_i$. The ratio between the voltage FWHM and the most probable value $U_{\max}$ of the resulting histograms of $U_i$ (i.e., the different α-spectra) was used to extract the relative energy resolution $\Delta U_i$, according to

$$\Delta U_i \% = 100 \times \frac{\text{FWHM}}{U_{\max}} \quad (3)$$

4. Results and discussion

The TCT measurements were successfully carried out in the range from room temperature up to $\approx 473$ K. The derived average pulse widths $t_c$ as well as the average integrated pulse areas $A_i$ for all temperature steps are compiled in Table 1, while a selection of reference pulse shapes (i.e., the average of all considered single pulses) are presented in Fig. 2. Good agreement is found for the herein presented measurements at room temperature with data obtained by others [7,20,25,26].

The shortest transit times, i.e., the fastest drift times, for both charge carriers are obtained at room temperature. With increasing temperature, the amplitude of the current responses gradually decreases at a simultaneous broadening of the pulse shapes (see Fig. 2). Reaching $\approx 473$ K, the signal traces start to divert from a flat-top profile to a distorted trapezoidal form with decreasing currents for electrons and increasing currents for holes. Following a similar argumentation as outlined in [20], this observation can be attributed to a superimposed effect of charge carrier trapping and a uniform positive space-charge concentration in the bulk. Further insights into trapped space charge and its probing can be found in [27,28]. The direct comparison of the drift times (see Table 1 and Fig. 3A) of both charge carriers reveals overall shorter drift times for holes within the investigated temperature range. At the same time, the temperature dependence is larger for holes than for electrons when going to higher sensor temperatures. Consequently, at roughly 465 K a crossing point of both progressions with temperature can be observed (see Fig. 3A). Following Eq. (2), inverse trends occur for the drift velocity $v_{dc}$ as a function of temperature (see Fig. 4) in comparison to Fig. 3A.

### Table 1

| Temperature [K] | $t_c$ [ns] | $A_i$ [pVs] | $c_{e-i}$ [eV] | $t_c$ [ns] | $A_i$ [pVs] | $c_{e-i}$ [eV] |
|----------------|-------------|-------------|----------------|-------------|-------------|----------------|
| 294            | 7.87(7)     | 597(15)     | 13.8(3)        | 294         | 5.70(5)     | 595(14)        |
| 333            | 8.31(8)     | 601(14)     | 13.7(3)        | 333         | 6.20(6)     | 590(14)        |
| 344            | 8.45(8)     | 603(13)     | 13.7(3)        | 344         | 6.62(6)     | 594(15)        |
| 355            | 8.61(8)     | 601(14)     | 13.7(3)        | 355         | 6.85(8)     | 591(14)        |
| 365            | 8.75(8)     | 600(13)     | 13.7(3)        | 365         | 7.10(8)     | 594(14)        |
| 383            | 9.02(9)     | 600(14)     | 13.7(3)        | 383         | 7.67(8)     | 582(13)        |
| 395            | 9.27(9)     | 601(10)     | 13.7(3)        | 395         | 7.99(9)     | 588(14)        |
| 405            | 9.46(10)    | 603(14)     | 13.7(3)        | 405         | 8.32(7)     | 581(14)        |
| 415            | 9.68(10)    | 605(14)     | 13.7(3)        | 415         | 8.69(8)     | 585(13)        |
| 425            | 9.93(11)    | 612(14)     | 13.5(3)        | 425         | 9.07(11)    | 584(12)        |
| 435            | 10.18(11)   | 608(16)     | 13.6(4)        | 435         | 9.45(11)    | 591(14)        |
| 445            | 10.47(11)   | 611(17)     | 13.5(4)        | 445         | 9.88(11)    | 593(14)        |
| 455            | 10.79(12)   | 605(13)     | 13.6(3)        | 455         | 10.37(14)   | 587(14)        |
| 465            | 11.10(13)   | 605(15)     | 13.6(3)        | 465         | 10.88(11)   | 593(12)        |
| 475            | 11.42(13)   | 607(14)     | 13.6(3)        | 475         | 11.46(15)   | 591(13)        |

Electrons

| $T$ [K] | $t_c$ [ns] | $A_i$ [pVs] | $c_{e-i}$ [eV] |
|---------|-------------|-------------|----------------|
| 294     | 7.87(7)     | 597(15)     | 13.8(3)        |
| 333     | 8.31(8)     | 601(14)     | 13.7(3)        |
| 344     | 8.45(8)     | 603(13)     | 13.7(3)        |
| 355     | 8.61(8)     | 601(14)     | 13.7(3)        |
| 365     | 8.75(8)     | 600(13)     | 13.7(3)        |
| 383     | 9.02(9)     | 600(14)     | 13.7(3)        |
| 395     | 9.27(9)     | 601(10)     | 13.7(3)        |
| 405     | 9.46(10)    | 603(14)     | 13.7(3)        |
| 415     | 9.68(10)    | 605(14)     | 13.7(3)        |
| 425     | 9.93(11)    | 612(14)     | 13.5(3)        |
| 435     | 10.18(11)   | 608(16)     | 13.6(4)        |
| 445     | 10.47(11)   | 611(17)     | 13.5(4)        |
| 455     | 10.79(12)   | 605(13)     | 13.6(3)        |
| 465     | 11.10(13)   | 605(15)     | 13.6(3)        |
| 475     | 11.42(13)   | 607(14)     | 13.6(3)        |

Holes

#### Fig. 2.
α-particle-induced current pulses for the electron (top) and the hole drift mode (bottom) in the temperature range from 294 K to 475 K; the applied detector potential is indicated in the top left corner of both graphs.

#### Fig. 3.
Results from individual fits to 100 traces per temperature step with (A) the drift time $t_c$ (FWHM, errors are smaller than the symbols) and (B) the integrated area $A_i$; for electrons (red, solid markers) and holes (blue, open markers); the lines in (A) are drawn to guide the eye, whereas the horizontal lines in (B) indicate the average value for electrons of 604(15) pVs (solid line) and for holes of 590(14) pVs (dashed line).
Meanwhile, the averaged areas of the integrated current pulses remain unchanged within the given confidence interval throughout all applied temperatures (see Table 1 and Fig. 3B), thus indicating constant charge collection. The averaged, collected charge for the holes is found to be systematically lower by \( \approx 3\% \). This may be attributed to the different zero-field mobilities and hence, different trapping probabilities at defects during the initial stage of charge generation, for electrons and holes, respectively. Based on the mean integrated area of the current response at room temperature, i.e., \( A_e(e^-) = 597(15) \) pVs or \( A_h(h^+) = 595(14) \) pVs, the electron–hole-pair creation energy can be derived. Thereto, the electronically calibrated gain of 9.7 pVs/IC of the C2-HV broadband amplifier as well as the above calculated average energy deposition of an \( \alpha \)-particle in the bulk of the scCVD diamond sensor have to be taken into account (see Section 2). The correspondingly deduced electron–hole-pair creation energy at room temperature amounts to \( \epsilon_e-h(e^-\text{-drift}) = 13.8(3) \) eV and \( \epsilon_e-h(h^+\text{-drift}) = 13.9(3) \) eV, respectively, both being in reasonable agreement with results obtained by others [29–33]. Additionally, the electron–hole-pair creation energies \( \epsilon_e-h \) for all temperatures up to \( \approx 473 \) K were calculated (see Table 1). As expected and following from the complete charge collection across all investigated temperatures (see Fig. 3B) the values stay constant within the given uncertainties. The extracted mean values of the collected charge from all measured current pulses at the different temperatures lead to mean electron–hole-pair creation energies of \( \epsilon_e-h(e^-\text{-drift}) = 13.6(3) \) eV and \( \epsilon_e-h(h^+\text{-drift}) = 14.0(3) \) eV.

The mean noise level (rms) during the TCT measurements stayed below 3 mV at all times. The measured signal amplitudes for the electron drift mode are in the range of \( \approx 100 \) mV at 293 K and \( \approx 60 \) mV at 473 K, which results in signal-to-noise ratios of \( \text{SNR} = 33 : 1 \) and \( \text{SNR} = 20 : 1 \), respectively. Hence, the SNR is sufficient for the conducted measurements across all applied temperatures.

5. \( \alpha \)-spectroscopic properties

During the \( \alpha \)-spectroscopic measurements (electron drift mode only), the pulse amplitude (i.e., the charge yield) as well as the resolution remained constant over the investigated temperature range (see Fig. 5). In consideration of the above mentioned electronically calibrated gain of 17.8 mV/IC for the used Cx spectroscopic amplifier (see Section 2), the derived average pulse height of 1137(7) mV over all conducted measurements translates to an induced charge of 63.9(4) fC. The resulting mean electron–hole-pair creation energy of \( \epsilon_e-h(e^-\text{-drift}) = 13.3(1) \) eV coincides with the above determined energies from the TCT measurements and agrees well with reported values by others [29–33].

For the resolution an average value of 1.24% at \( E_{\alpha} = 5304 \) keV (i.e., 64 keV) was obtained. In the following, different contributions to the spectroscopic resolution are discussed. Hereto, the lower limit for the total noise level (see dotted line in Fig. 5B) was estimated from the individual noise contributions of the DAQ system, the spectroscopic amplifier, the intrinsic ionization fluctuation in the scCVD diamond sensor itself, and of the metalization [33]. The effective quantization noise from the 8.5 – 11-bit vertical resolution of the oscilloscope of 21 keV (FWHM) together with the ionization fluctuation within the sensor of 12 keV (FWHM), and the energy uncertainty due to self-absorption in the reference \( \alpha \)-source of 8.0 keV (FWHM) are all rather small. Larger contributions arise from the spectroscopic amplifier together with the relatively long electrical connection to the sensor unit, amounting to 33 keV (FWHM), as well as from the energy straggling of the \( \alpha \)-particles after passing through the metal layers of the electrode contributing with another 34 keV (FWHM). Taking into account all above mentioned contributions, the lower limit for the FWHM resolution amounts to 53 keV or 1.01% at \( E_{\alpha} = 5304 \) keV. Thus, the observed resolution is only \( \approx 25\% \) larger, than what we can account for from the listed noise contributions.

The herein presented measurements at high temperatures are in full agreement with earlier investigations, where the authors observed similar energy resolutions in their respective \( \alpha \)-spectra [3,12–15].

6. Conclusions

The charge carrier properties were investigated in the temperature range from room temperature up to \( \approx 473 \) K. The obtained increasing drift times \( t_{\text{drift}} \) during the TCT measurements and the thereof calculated decreasing drift velocities \( v_{\text{drift}} \), show that electrons are less affected by an increasing sensor temperature compared to holes. Distorting pulse shapes at the highest applied sensor temperature indicated a positive space charge built-up within the bulk of the scCVD diamond sensor.
The integrated pulse areas, i.e., the total collected charge, stayed constant throughout all applied temperatures. The calculated electron-hole-pair creation energies of $\epsilon_{\alpha}(E)$ from the integrated current responses at room temperature compare well with literature and the later on derived value of $\epsilon_{\alpha}(E)$ during the $\alpha$-spectroscopic measurements (a summary of the obtained results is given in Table 2).

The noise level remained constant during all measurements. This is furthermore supported by the observed stable $\alpha$-spectroscopic resolution across all applied temperatures with an average value of 1.24% at $E_0 = 5304$ keV, being in good agreement with results obtained by others.

The herein presented results render scCVD diamond-based detectors ready to be safely operated in HT $\alpha$-spectroscopic applications up to temperatures of at least 473 K. These measurements will be continued, using the same setup, but a generally improved HT sensor enclosure and electrical contacting. For this purpose, a ceramic-based PCB-structure is foreseen. These adjustments will allow for an investigation of different electrical contacting. For this purpose, a ceramic-based PCB-structure is foreseen. These adjustments will allow for an investigation of different electrical contacting.

CRediT authorship contribution statement

Benjamin Kraus: Investigation, Validation, Formal analysis, Writing - original draft, Visualization. Patrick Steinegger: Investigation, Software, Conceptualization, Methodology, Validation, Formal analysis, Writing - review & editing, Visualization, Project administration. Nikolay V. Aksenov: Investigation, Funding acquisition. Rugard Dressler: Software, Validation, Formal analysis. Robert Eichler: Conceptualization, Supervision, Funding acquisition. Erich Griesmayer: Investigation, Conceptualization, Methodology, Formal analysis. Dominik Herrmann: Methodology, Resources. Andreas Türler: Supervision, Funding acquisition. Christina Weiss: Investigation, Conceptualization, Methodology, Formal analysis.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Table 2

Summary of results concerning the electron-hole-pair creation energy, as obtained during the TCT measurements as well as from the $\alpha$-spectroscopic measurements, with $Q_{\text{coll}}$, the collected charge at room temperature, $\epsilon_{\alpha}$, the electron-hole-pair creation energy at room temperature, and $\overline{\epsilon}_{\text{alpha}}$ as well as $\overline{\epsilon}_{\text{alpha}}$, the mean values across all investigated temperatures; for comparative reasons the most recent value for $\epsilon_{\alpha}$ is given [33].

| TCT          | $Q_{\text{coll}}$ | $\epsilon_{\alpha}$ | $\overline{\epsilon}_{\text{alpha}}$ |
|--------------|-------------------|----------------------|--------------------------------------|
| Electrons    | 61.5(1) fC         | 13.8(3) eV           | 13.6(3) eV                            |
| Holes        | 63.4(4) fC         | 13.9(3) eV           | 13.6(3) eV                            |
| Electrons    | 62.5(1) fC         | 13.9(3) eV           | 13.6(3) eV                            |
| Holes        | 64.6(4) fC         | 14.0(3) eV           | 13.6(3) eV                            |

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