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# X-ray Beam Induced Current analysis of CVD diamond detectors in the perspective of a beam tagging hodoscope development for hadrontherapy on-line monitoring.

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**Keywords** CVD diamond, X-rays detection, Hadrontherapy monitoring

## Highlights

- Diamond double stripped beam monitor prototypes for hadrontherapy monitoring.
- Chemical Vapor Deposited diamond detector performance evaluated under X-rays.
- 2D current maps evaluated on diamond metallized surface using X-rays micro beams.
- Time resolution and bunch detection efficiency at contact strip crossing points.

## Abstract

The intrinsic electronic properties of diamond make it suitable for radiation-hard and very fast detector development with good signal to noise ratios. With the advent of new generations of ion accelerators either for physics (nuclear and high energy physics) or medical applications (hadrontherapy and synchrotron radiation radiotherapy) there is a need for a very accurate beam monitoring in high radiation environments. Diamond is particularly suited to these applications. Fast pulse detection mode for time stamp, and current integration mode for operation as beam monitors at high particle rates are targeted. Commercial single-crystal, polycrystalline and heteroepitaxial diamonds produced by Chemical Vapor Deposited (CVD) method are analyzed and compared by means of X-ray Beam Induced Current (XBIC). Their performance as particle detectors is investigated using a 8.5 keV X-ray photon micro-bunch beam at ESRF (European Synchrotron Radiation Facility). This facility provides a focused ( $\sim 1 \mu\text{m}$ ) pulsed beam (100 ps bunch duration), producing an almost uniform energy deposit along the beam irradiated volume in the detector, therefore closely mimicking the interaction of single charged particles. The XBIC set-up of the ID21 beamline enabled us to draw 2D response maps of detectors with disk- and strip metal contact patterns. Using the pulse-synchronized XBIC measurements, a time resolution of 150 ps RMS and bunch detection efficiency of  $\sim 100\%$  were evaluated at the contact strip crossing points of a first prototype polycrystalline beam monitor.

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1 **1. Introduction**

2

3 Compared to other semiconductor detectors, diamond-based detectors exhibit several advantages. A  
4 high resistivity ( $>10^{13} \Omega \text{ m}$ ) due to its wide electronic band gap (5.48 eV) results in an almost negligible  
5 leakage current and the low dielectric constant ( $\sim 5.72$ ) in practice results in lower preamplifier noise  
6 levels as compared to silicon devices of similar geometry. The high diamond mobility for both charge  
7 carriers [1] also results in a very fast detector response, enabling tens of picoseconds time resolution and  
8 high count-rate capabilities [2]. Diamond detector applications are therefore more and more considered  
9 in various fields, such as high-energy and nuclear physics [3], and medical applications [4], [5], [6], [7],  
10 [8].

11 The development of new generations of ion accelerators either for physics (nuclear and high-energy  
12 physics) or medical applications (hadrontherapy and synchrotron radiation radiotherapy) generates the  
13 need for a very accurate beam monitoring with precise and fast counting in these high radiation  
14 environments. Diamond is well suited to these areas of application [8], [9], [10], [11], [12], [13].

15 The development of a diamond-based beam monitor is part of a large-scale French experimental project  
16 emanating from the national collaboration CLaRyS and intended for online monitoring of hadrontherapy  
17 [14]. Hadrontherapy is an alternative cancer therapy that consists in the treatment of tumors by light ion  
18 beams (e.g. carbon or protons). The ions deposit a large fraction of their dose at the end of their path, in  
19 the so-called Bragg peak. As compared with conventional photon radiotherapy, this allows a more  
20 efficient dose delivery in the tumor, with a reduction of the dose deposited in the nearby healthy organs.  
21 The conformation of the deposited dose to the tumor volume is provided by distributing spread-out  
22 Bragg peaks in the target volume, either spot by spot, or by means of passive scattering and energy  
23 degradation. However, since multiple sources of uncertainty in the ion range may cause deviations from  
24 the planned dose distribution [15], online control of the ion range is desired in order to improve safety  
25 margins and optimize the ballistic advantage of ion therapy.

26 During an irradiation with ion beams, nuclear reactions occur for a fraction of the incident projectiles.  
27 Photons in the range 1-10 MeV are emitted almost isotropically within a picosecond after such reactions.  
28 It has been shown experimentally that the longitudinal distribution of such prompt-gamma production  
29 is highly correlated to the primary ion range [16], [17], [18].

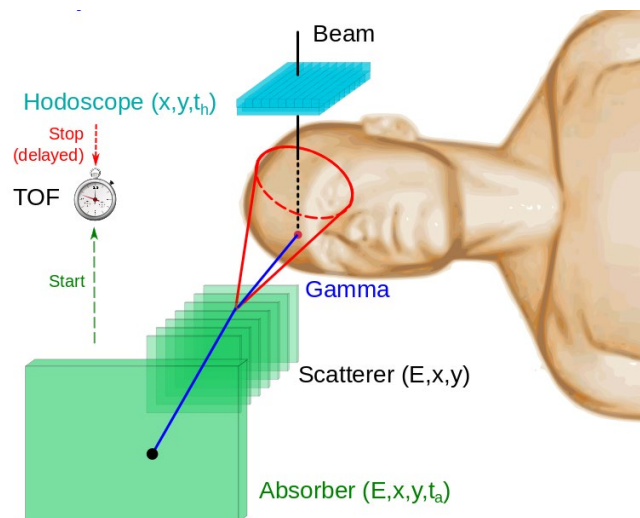


Figure 1 Scheme of the combination of a Compton camera and a beam hodoscope for Time of Flight prompt-gamma range verification.

30 Based on these observations, two systems for prompt gamma detection are currently under development  
31 in the CLaRyS collaboration [19], [14]: a collimated gamma camera, and a Compton camera (Figure 1).  
32 The originality of the two systems is the use of Time of Flight (ToF) in order to reject the background  
33 induced by secondary particles. Depending on the beam time-structure and intensity, a beam tagging  
34 hodoscope may be necessary to detect the arrival times of ion bunches or even individual projectiles.  
35 This hodoscope will be also used for transverse position measurement, giving access to 3D prompt-  
36 gamma imaging. Thus the hodoscope plays a major role in all the detection concepts, and has to fulfil  
37 the requirement of providing both position and timing information of the incident hadron beam at about  
38 100 Mcps count rates. As an alternative to a large-area scintillating-fiber hodoscope already under  
39 construction [14], a diamond-based hodoscope is being developed which presents the advantages of fast  
40 response and high radiation tolerance. The device must operate in hadrontherapy, in a harsh radiation  
41 environment with a long operational lifetime, withstanding radiation damage. The strong covalent  
42 bonds, large band gap and small neutron cross section all contribute to making diamond a radiation hard  
43 material [4], [20], [21]. The radiation hardness of diamond compared to that of the widespread silicon  
44 technology can be demonstrated by the SRIM simulation software. It was already shown that the number  
45 of vacancies generated by an incident carbon ion beam of 400 MeV/u (a typical carbon beam energy  
46 used in hadrontherapy) is only 1300 per incident ion in diamond, compared to 3500 in silicon [22].  
47 Reference [23] describes the behaviour of sCVD and pCVD diamond detectors as irradiated by 70 MeV  
48 and 800 MeV protons for fluences up to  $\sim 10^{16}$  p/cm<sup>2</sup>. A significant decrease of the charge-trapping  
49 mean free path was observed in sCVD diamonds at such fluences, with a less pronounced effect  
50 observed for pCVDs. However, such fluences correspond to several years of operation in clinical  
51 conditions.

52 This diamond beam monitor will be equipped with the largest available sensors. The detectors will be  
53 segmented with double side orthogonal metallic strips readout (X and Y directions), in order to provide  
54 spatial resolution while maintaining low detector capacitance values. Diamonds will be assembled in a  
55 mosaic arrangement to cover the needed detection area (typically 1 cm<sup>2</sup> or 4 cm<sup>2</sup>). Finally, a further  
56 specific goal of the whole project is the development of dedicated readout electronics (fast preamplifier,  
57 current integration) using designs based on both discrete and integrated components (CMOS 130 nm  
58 ASICs), and a data acquisition system [24] which is compliant with the CLaRyS  $\mu$ TCA acquisition  
59 system [25]. The current integration front-end electronics must satisfy the requirements of the beam  
60 time structure and ensure a linear response to the beam intensity variation over a wide dynamic range.  
61 Using a 500  $\mu$ m thick single crystal Chemical Vapor Deposited (sCVD) diamond detector, this  
62 represents a charge ranging typically from 7.5 fC for protons of 70 MeV, up to 520 fC for carbon ions  
63 of 95 MeV/nucleon, in a pulse of some nanoseconds, regards to the high mobility of charge carriers in  
64 diamond.

65 Commercial CVD single-crystal, poly-crystalline and heteroepitaxial diamonds were acquired. These  
66 were processed and characterized with short-bunched 8.5 keV photons from the European Synchrotron  
67 Radiation Facility (ESRF). Third generation Synchrotron facilities offer the capability of highly focused  
68 beams, to  $<1$   $\mu$ m FWHM. As regards energy deposition in the diamond, in the ESRF 4-bunch machine  
69 mode, the X-ray pulses are of  $\approx 100$  ps duration spaced at 704 ns intervals, and thus mimic the passage  
70 of single ionizing particles. Indeed, their exponential absorption length  $1/\mu \sim 750$   $\mu$ m [26] results in an  
71 energy deposit which is almost uniformly distributed over the whole thickness of our samples (300 -  
72 500  $\mu$ m). We characterized the temporal and charge/current signal responses of several diamond  
73 detectors over a large fraction of their surfaces. The possible application of diamonds as a position  
74 sensitive detector is discussed in the present paper from these radiation test results. The respective  
75 performances in terms of surface detection homogeneity of large area ( $\sim 1$  cm<sup>2</sup>) polycrystalline- and  
76 heteroepitaxially-grown diamonds are compared to single-crystal diamond detectors. Finally, an X-ray  
77 analysis of a double side striped polycrystalline diamond surface equipped as first beam monitor  
78 prototype was performed. Diamond detector performances were analyzed both in current integration

79 and in pulsed modes. The X-ray detection efficiencies and the time resolutions were evaluated at various  
80 top-bottom strip crossing points.

## 81 2. Material and methods

### 82 2.1 Diamond sensors

83 CVD synthetic diamonds are grown by the technique of microwave plasma-enhanced CVD [27]. Single  
84 crystal sCVD diamonds are almost free from defects acting as charge traps, but their available area is  
85 restricted  $1\text{ cm}^2$  ( $1\text{ cm}^2$  is still considered as an upper limit, not available on-shelf). Polycrystalline pCVD  
86 diamonds present grain boundaries and stacking faults, structural defects which act as undesirable charge  
87 trapping centers for free charge carriers. The main interest in pCVD material is that it can be grown as  
88 wafers to sizes  $> 6$  inches [28]. Lastly, heteroepitaxially grown diamonds, hereafter referred to as  
89 Diamond On Iridium (DOI), were tested. Such diamonds are also grown as wafers, with areas  $> 1\text{ cm}^2$   
90 cut from a parent wafer after substrate removal [29], [30].

91 In the present study, we used both sCVD and pCVD diamonds from Element6 [28], and heteroepitaxially  
92 grown DOI diamonds from Audiatic and the University of Augsburg [29], [30]. The samples tested  
93 ranged from  $300\text{ }\mu\text{m}$  to  $500\text{ }\mu\text{m}$  in thickness, and with areas from  $0.45 \times 0.45\text{ cm}^2$  to  $1 \times 1\text{ cm}^2$ . The  
94 suitability of these diamonds as detectors was first investigated by measuring their current voltage  
95 characteristics, as illustrated by Figure 2. Similar measurements were made previously [31] [32], carried  
96 out at room temperature and ambient pressure. The detectors were in darkness with electromagnetic  
97 shielding, with a bias voltage applied to one side of the detector. The current generated by the detector,  
98 without external excitation other than the electric field created by the bias voltage, was measured from  
99 the opposite, ground potential side using a Keithley 6487 picoammeter, of minimum resolution of  $\pm 10$   
100 fA (for a range of 2 nA). All the diamonds thus tested had aluminum contacts except (and only for these  
101 preliminary tests) the sample DOI-1 (which was not metallized) and a  $300\text{ }\mu\text{m}$  thick pCVD sample  
102 (which was contacted with silver lacquer). Except for DOI-1, all the diamonds showed an asymmetry in  
103 their leakage current measurements for positive and negative applied bias voltage. This asymmetric  
104 behavior revealed non Ohmic contacts that showed rather Schottky-like behaviour. The observed  
105 asymmetry probably resulted from the choice of the aluminum layer deposited on the diamond top and  
106 bottom sides as in [33] and variations in the diamond surface preparation made before the contact  
107 metallization. Note also that the values of the leakage currents were several orders of magnitude higher  
108 on the DOI samples (except for DOI-2) compared to those of pCVD. This observation may indicate, *a*  
109 *priori*, evidence of difference in crystal quality between the various detectors and that is discussed later  
110 in this paper. Aluminum contacts have already proven their efficiency for detectors of traversing ionizing  
111 particles and for X-rays [33] [34]. Contacts such as TiPtAu are known to exhibit a more ohmic behaviour  
112 [35], but they were not used in our tests in order to minimize soft X-ray absorption in the contacts  
113 themselves [36].

114 These leakage current measurements enabled us to characterize the response of the diamond detectors  
115 in pulse mode and define an optimal bias for the XBIC experiments. To estimate their charge collection  
116 efficiencies, the diamond detectors were also characterized with a  $^{241}\text{Am}$  5.5 MeV alpha particle test  
117 bench. A detailed study, reported in [37], concludes that the  $0.45 \times 0.45\text{ cm}^2 \times 500\text{ }\mu\text{m}$  sCVD sample  
118 exhibited an overall charge collection efficiency of  $\approx 100\%$  as compared to  $\approx 30\%$  for the  $1 \times 1\text{ cm}^2 \times$   
119  $300\text{ }\mu\text{m}$  pCVD sample, and  $\approx 40\%$  for the  $0.5 \times 0.5\text{ cm}^2 \times 300\text{ }\mu\text{m}$  DOI sample from the University of  
120 Augsburg. This last observation is indicative of the density of defects in the crystal structure of the  
121 diamonds tested and also reveals the overall behavior of the detectors under exposure to weakly  
122 penetrating ionizing particle radiation.

123  
124

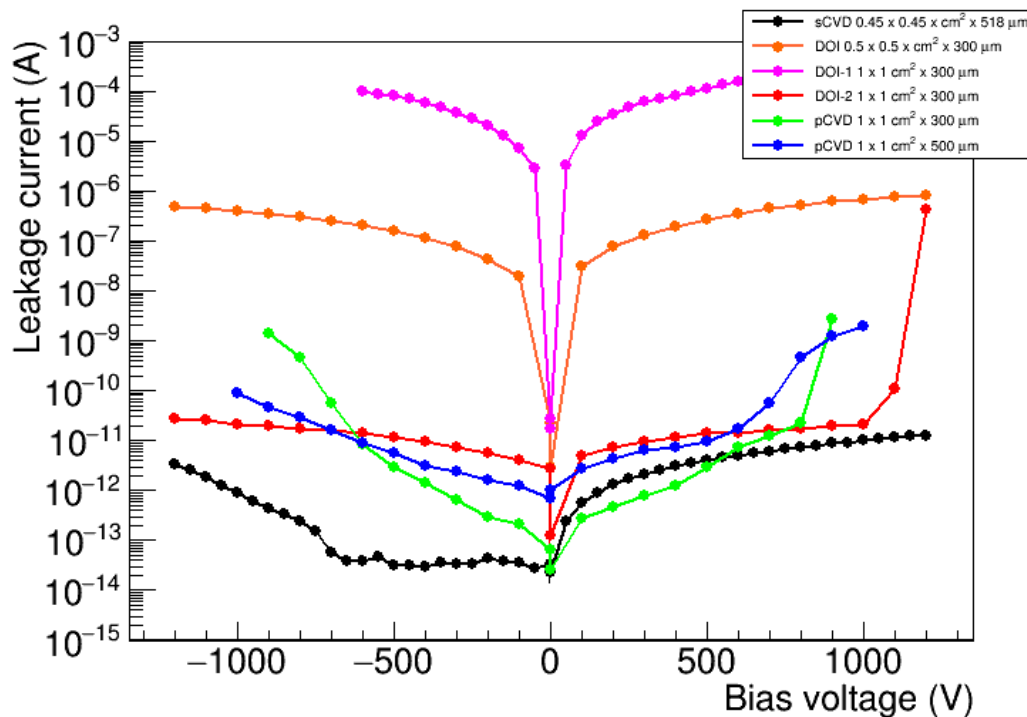


Figure 2: Leakage currents versus the bias voltage. All the diamond samples exhibit aluminum contacts except DOI-1 (not metallized) and the 300  $\mu\text{m}$  thick pCVD (metallized with silver lacquer).

125 **2.2 Detector processing**  
 126

127 The aluminum disk shaped metallization was locally performed using Distributed Microwave Plasmas  
 128 (DMW) deposition, a technology developed at LPSC [38]. The sensor contact, as detailed in references  
 129 [31], [32] for the disk-shaped metallization consists of a 50 nm thick aluminum layer deposited on both  
 130 sides. The diamond is sandwiched between two printed circuit boards (PCB). A plastic spacer is used to  
 131 avoid damage to the diamond while the two PCBs are screwed together. The electrical contact is ensured  
 132 by a copper ring which surrounds a window opening in the PCB that reveals the contacted diamond.  
 133 The diamond holders provide direct connection to 50  $\Omega$  impedance SMA connectors. These enable  
 134 reversible bias and signal readout connections to either side of the diamond. At ESRF, the detector  
 135 holder was also enclosed in an electromagnetic-shielding box (Figure 3 (a)) with kapton tape covered  
 136 apertures for the X-ray beam passage.

137 Three hodoscope prototypes were built. One was equipped with a pCVD diamond and two with DOI  
 138 diamonds (all were  $1 \times 1 \text{ cm}^2 \times 300 \mu\text{m}$  in size). Each diamond was equipped with a unique, double-  
 139 side strip metallized diamond sensor [32]. The strips on opposing sides are arranged orthogonally,  
 140 enabling X and Y spatial localization. The lift-off process was used to produce the strips. It consists in  
 141 creating structures (patterning) using a sacrificial material (e.g., photoresist). This was performed at the  
 142 NANOFAB laboratory as described in reference [32]. The resulting metallization on the diamond sensor  
 143 consists of 8 strips of 950  $\mu\text{m}$  width separated by a 100  $\mu\text{m}$  gap, surrounded by a guard ring (see Figure  
 144 5 (a) and Figure 5 (b) for the detailed schematic). Only one DOI diamond exhibited a non-regular size  
 145 gap (Figure 5 (b)). The choice of the guard ring is based on the desire to reduce edge effects, which is  
 146 essential for this type of detector intended for spatial localization.

147 Unlike the case of the disk-shaped metallization, wire bonds ensured the electrical connection between  
 148 each diamond strip and the PCB. This led us to make a thicker deposit of aluminum for the strip  
 149 electrodes to avoid contact tearing when placing the bonding wires. In addition, a set of discrete current  
 150 amplifiers designed at LPSC (1 GHz bandwidth with 30 dB gain) were mounted on the circuit board.  
 151 The entire assembly was placed inside an electromagnetic shielding box provided with SMA connectors  
 152 to enable biasing and analog signal readout for each strip (Figure 3 (b)).

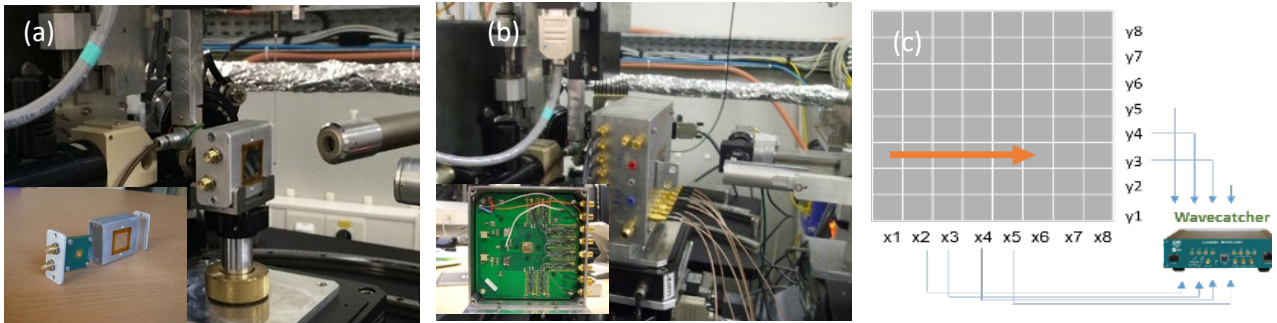


Figure 3: (a) Detector holder for disk metallized diamond and (b) the 1 cm<sup>2</sup> double side striped pCVD diamond mounted on the PCB inside respectively in their electromagnetic shielding box and under test on the ID21 beam line at ESRF. (c) details for 1cm<sup>2</sup> pCVD detector beam scanning scheme of the tracks with signal readout using the Wavecatcher acquisition system.

153

### 154 2.3 XBIC experimental set-up

155 The diamond detectors operate as solid state ionization chambers. The bias voltage (typically 1V/ $\mu\text{m}$ )  
 156 is applied on one side of the diamond and the signal readout is performed on the opposite side which is  
 157 at ground potential. The shielding box containing the diamond holder that corresponds to either the disk  
 158 or striped metallized samples was attached to a magnetic kinematic mount, which provided micron  
 159 positioning reproducibility at the sample position of the micro-diffraction end station (in air) of the ID21  
 160 beamline at ESRF (Figure 3 (a) and (b)).

161 The diamond detectors were placed in between two Si p-i-n diodes used for beam intensity normalization  
 162 purposes as illustrated on the schematic drawn in Figure 4. The diode located upstream of the diamonds  
 163 relative to the beam is denoted as Iodet. Iodet is a diode with a central hole to enable beam passage. This  
 164 diode is excited by X-rays scattered by a thin Ti layer deposited on a Si<sub>3</sub>N<sub>4</sub> foil placed just upstream.  
 165 The diode collects the fluorescence of Ti and gives a signal proportional to the incident intensity. The  
 166 diode located downstream is denoted as Idet. Two different data running modes were used during the  
 167 whole experiment: current integration mode (diamond signal currents measured with electrometers) and  
 168 the signal pulse readout mode (diamond signal into 50  $\Omega$  input impedance fast amplifiers).

169 To draw current maps in current integration mode, three Keithley 485 picoammeters were used (named  
 170 Pico 3 and Pico 1 for those connected respectively to Iodet and Idet, and K485 for those connected to  
 171 the diamond samples). Each Keithley analog voltage output was coupled to a voltage-to-frequency  
 172 converter read out by a standard ESRF counter board. This system was configured to provide 100 ms  
 173 current integration periods that could be synchronized to position scans of the diamonds in the X-ray  
 174 beam. For the signal pulse readout mode, the disk metallized diamond signal output was connected to a  
 175 CIVIDEC C2 low-noise broadband amplifier (2 GHz, 40 dB from CIVIDEC Instrumentation Company  
 176 [39]). This exhibits a 50  $\Omega$  input impedance and is designed to work with FWHM pulse widths of less  
 177 than 1 ns. The signal measurements were measured alternatively with a 500 MHz, 3.2 GS/s digital  
 178 sampling 'WaveCatcher' [40] system, and with a 2 GHz, analog bandwidth 20 GS/s Digital Sampling  
 179 Oscilloscope (DSO LeCroy 620Zi) [41]. These systems could be configured for readout by the ID21

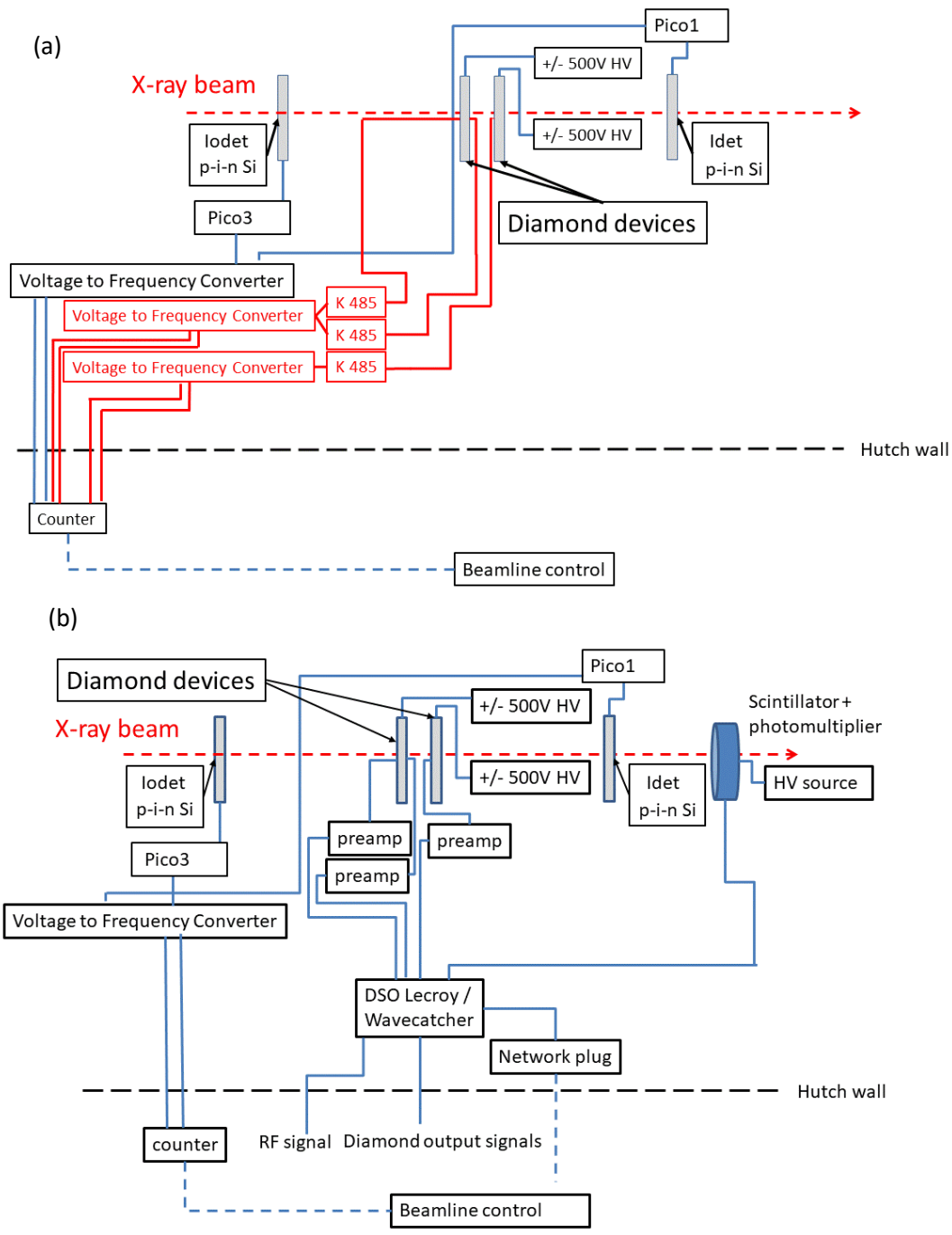


Figure 4 Schematic of the diamond devices set-up at ESRF on ID21 beam line in current mode (a) and in pulse mode (b). Iodet is a diode with a central hole to enable beam passage.

180 beamline data acquisition system in a continuous acquisition mode, recording a set of waveforms, thus  
 181 enabling large data statistics for offline analysis. In signal pulse readout mode, the timing of the data  
 182 acquisition for each pulse readout was precise at a level  $<1$  ns with respect to the incidence of the X-ray  
 183 beam pulses on the diamonds: this was achieved by trigger-locking the acquisition systems to a reference  
 184 signal that was a sub-multiple of the ESRF synchrotron 352 MHz radiofrequency. The DSO and  
 185 Keithley 485 acquisitions were both fully integrated into the ID21 SPEC software acquisition framework



186 which enabled us to measure the response of the detectors as they were motor-scanned across the beam.  
 187 Furthermore, the 8 channels readout available on the WaveCatcher made it possible to record 4 strips in  
 188 X and 4 strips in Y while the first diamond-based beam monitor prototype was motor scanned across  
 189 the beam (Figure 3 (c)); and thus evaluate its beam detection efficiency and time resolution performance  
 190 at the precision level of the X-rays ID21 micron-sized beam, focusing on specific diamond areas of  
 191 interest which could be selected by the analysis of previous area- or line- scans made with the Keithley  
 192 current mode set-up.

193 **2.4 Summary of the diamond samples tested at ESRF versus data acquisition set-up and data**  
 194 **analysis mode.**

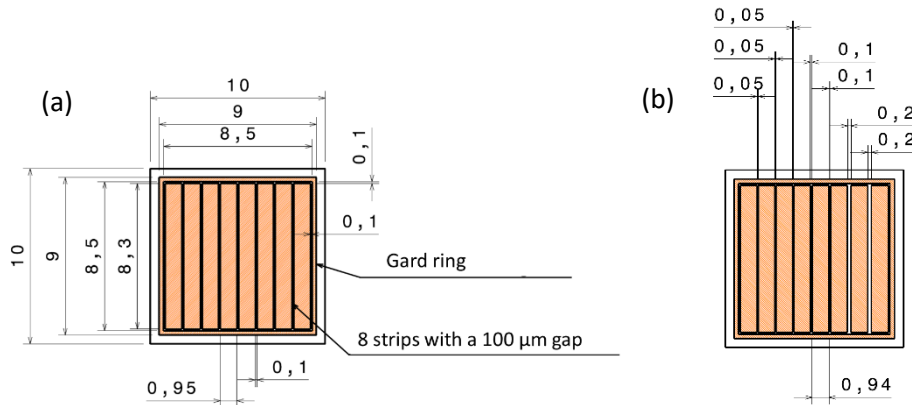


Figure 5 Details of the 8 strips distribution over the tripped diamond surface in the case of the regular spacing (a) and the non-regular one (b).

Diamond Material	Manufacturer	Electrode	Read-out amplifier	Data acquisition	Acquisition mode
sCVD 0.45 × 0.45 cm <sup>2</sup> × 518 µm	Element 6	Disk	CIVIDEC C2	Lecroy DSO	Pulse mode
pCVD 1 × 1 cm <sup>2</sup> × 500 µm	Element 6	Disk	none	Keithley	Current integration
pCVD 1 × 1 cm <sup>2</sup> × 300 µm	Element6	8 strips with regular spacing	none	Keithley	Current integration
			LPSC	Wavecatcher	Pulse mode
DOI 0.5 × 0.5 cm <sup>2</sup> × 300 µm	Augsburg University	Disk	none	Keithley	Current integration
DOI-1 1 × 1 cm <sup>2</sup> × 300 µm	Audiatec	8 strips with regular spacing	none	Keithley	Current integration
DOI-2 1 × 1 cm <sup>2</sup> × 300 µm	Audiatec	8 strips with non-regular spacing	none	Keithley	Current integration

Table 1 Summary of the various diamond detector configurations used

196 A summary of the diamond samples tested during the XBIC experiment is presented in Table 1. In  
 197 Figure 5 (a) the details of the regular 8 strips distribution on the surface for the pCVD and DOI-1  
 198 diamond  $1 \times 1 \text{ cm}^2 \times 300 \text{ }\mu\text{m}$  samples are presented. On DOI-2 ( $1 \times 1 \text{ cm}^2 \times 300 \text{ }\mu\text{m}$ ) Figure 5 (b)  
 199 the strip intervals vary from  $50 \text{ }\mu\text{m}$  to  $200 \text{ }\mu\text{m}$ . DOI-2 has been metallized with strips spaced with a  
 200 variable pitch to assess the load sharing capacities between two neighboring strips.

201 One of the first objectives of the X-ray beam test was to check the homogeneity of each diamond  
 202 response over its whole surface and to compare detector performances versus diamond growth process.  
 203 To do this, a calibration technique in current mode was developed to make a surface mapping  
 204 comparison between the different diamond samples (sCVD, pCVD and DOI, see Table 1).

## 205 2.5 Surface mapping procedure in current integration mode

206

### 207 a) X-ray flux calibration.

208

209 Beam flux measurements were made at the very beginning of the experiment when no diamond  
 210 sample was placed in between the two p-i-n diodes. With the 8.5 keV X-ray beam turned on,  
 211 the beam flux  $\Phi(\text{ph})$  measured in number of photons per seconds (ph/s) can be calculated as  
 212 follows:

213

$$214 \quad \Phi(\text{ph}) = (I_{\text{pico1}} - I_{\text{darkpico1}}) \times \frac{E_{\text{eh/Si}} \times \text{Times}}{E_{\text{ph}} \times e \times T_0}, \quad (1)$$

215

216 where:

- 217 •  $I_{\text{pico1}}$  is the current measured on the Keithley 485 picoammeter connected to the Idet p-i-n diode (A),
- 218 •  $I_{\text{darkpico1}}$  is the value of  $I_{\text{pico1}}$  measured when the beam is off (380 pA),
- 219 •  $E_{\text{eh/Si}}$  is the energy to create electron-hole pairs in silicon ( $\sim 3.6 \text{ eV}$ ),
- 220 • Times is the time measured in seconds during which a data acquisition was done at a given beam  
 221 position on the sample surface
- 222 •  $T_0$  is the reference time used for a normalization purpose ( $T_0=1 \text{ s}$ ),
- 223 •  $E_{\text{ph}}$  is the X-ray energy (8.5 keV),
- 224 •  $e$  is the electron charge ( $1.602 \cdot 10^{-19} \text{ C}$ ).

225

226 In the 4 bunch operation mode of the ESRF synchrotron, the nominal value of the injected synchrotron  
 227 ring current was 32 mA. However, typically during a given period of 20 minutes, this current decreased  
 228 quasi-exponentially to reach  $\sim 20 \text{ mA}$  before a beam refill was carried out over few minutes to again  
 229 reach the initial value of 32 mA, and so on for the entire duration of a data acquisition run. When the  
 230 ESRF synchrotron current was at its maximum 32 mA we measured  $I_{\text{pico1}} = 6.7 \cdot 10^{-7} \text{ A}$ , and from (1), we  
 231 calculated  $\Phi_{32\text{mA}}(\text{ph}) = 1.79 \cdot 10^9 \text{ ph/s}$ . This represents 1340 photons in a single bunch.

232

233

234 **b) X-ray current calibration.**

235

236 For each diamond sample motor-scanned in the beam, three surface maps were recorded simultaneously:  
237 one for each p-i-n diode, and one for the diamond sample itself. The diode located upstream is not placed  
238 directly in the beam but detects scattered photons. One would expect from the recorded map a very  
239 homogeneous data distribution over the whole surface. All the points of each map were stored in  $n \times p$   
240 matrices, where  $n$  and  $p$  are respectively the row and column numbers in the 2D maps. The calibrated  
241 matrix for the diamond sample is measured in nA units, corrected (i.e. normalized) for the time  
242 decreasing beam intensity between the synchrotron storage ring refills as described in 2.5 a).

243 And, finally, a transmitted map  $M_{\text{transmitted}}$  has been drawn using the formula:

244 
$$M_{\text{transmitted}} = \frac{I_{\text{ph-out}}}{I_{\text{ph-in}}}. \quad (2)$$

245 Where  $I_{\text{ph-out}}$  and  $I_{\text{ph-in}}$  are the matrix of current values measured on each p-i-n diode situated respectively  
246 downstream and upstream the diamond sample.

247

248 **2.6 Surface mapping procedure in the pulse readout mode**

249

250 **a) Signal recording and mean current calculation**

251

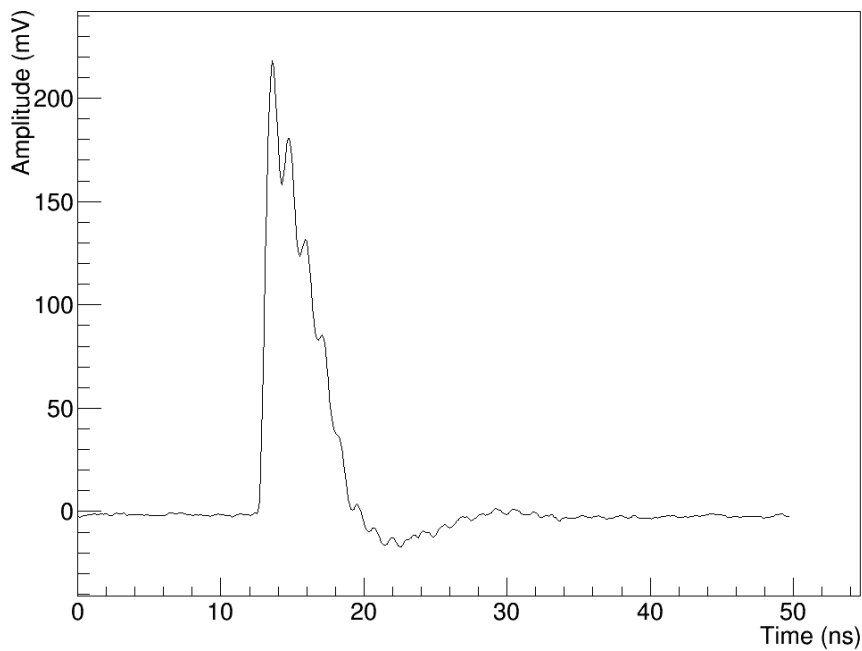


Figure 6 sCVD signal waveform recording using the DSO set-up (500 time points separated by 100 ps).

252 The diamond samples were also motor-scanned across the beam while their output signals were recorded  
253 using the Lecroy DSO set-up. This technique enabled us to perform simultaneously current mapping

254 (although less precise by comparison with the current mode described previously) and diamond response  
 255 waveform analysis (the main objective of this type of mode). The acquisition window was optimized  
 256 and fixed for the entire duration of data recording. Average waveform time trace (Figure 6 for sCVD  
 257 output signal) was evaluated over 64 DSO triggers by the DSO itself, and this averaged trace consisting  
 258 of 500 time points spaced by 100 ps read-out by the data acquisition system and stored for an offline  
 259 analysis purpose at each point of the diamond surface. Taking as an example the case of the sCVD  
 260 diamond with a surface of  $0.45 \times 0.45 \text{ cm}^2$ , a total of 8281 points spaced  $50 \mu\text{m}$  apart were recorded to  
 261 create the surface map of the diamond sample. An offline procedure was used to calculate the average  
 262 value of the current,  $I_{\text{mean}}$  at each position point as follows: the baseline of each signal is calculated on  
 263 the first 50 bins of each waveform time trace just prior to the arrival time of the X-ray pulse. The quantity  
 264  $Q$  of charges measured is deduced from calculation of the signal integrated over the entire recorded trace  
 265 period with the baseline subtracted value. The average current intensity measured in nA units in each  
 266 point of the map is then calculated as follows:

$$267 \quad I_{\text{mean}} = \frac{Q}{T_{\text{beam}}} = \frac{\sum_i V_i \times t_{\text{bin}}}{R \times \text{Gain} \times T_{\text{beam}}}, \quad (3)$$

268 where

- 269 •  $V_i$  is the signal voltage value after baseline subtraction, measured at bin number  $i$  on the  
 270 waveform distribution recorded with the Lecroy DSO set-up,
- 271 •  $t_{\text{bin}}$  is the value in nanoseconds of the time scale on the Lecroy DSO set-up for the waveform  
 272 data acquisition,
- 273 •  $R$  is the value of the input impedance ( $50 \Omega$ ),
- 274 • Gain is the nominal value of the preamplifier gain, in the present experiment CIVIDEC C2 ones  
 275 are concerned, we use the nominal value of 40 dB given by that manufacturer, although we did  
 276 not measure this for each preamplifier,
- 277 •  $T_{\text{beam}}$  relies on the beam delivery time in the very specific 4 bunch mode at ESRF and is equal  
 278 to 704 ns, which corresponds to the beam intervals between two consecutive beam pulses.

279

280 ***b) ESRF beam current calibration.***

281

282 As explained above, in the 4 bunch operation mode of the ESRF, the initial value of the synchrotron  
 283 storage ring current during the present experiment was 32 mA. During a given measurement period, the  
 284 current value decreases before a beam refill is carried out (refills occurred at intervals ranging from 20  
 285 to 60 minutes), to reach again the initial current value of 32 mA, and so on for the entire duration of data  
 286 acquisition. Unfortunately, it was not possible to record precisely the beam intensity variations in time  
 287 necessary to normalize the recorded diamond output signals. Consequently, such maps are not  
 288 normalized to the beam intensity.

289

290 **3. Results and discussion of the performances of various diamond samples in current mode.**

291

292 **3.1 Single crystal diamond (sCVD).**

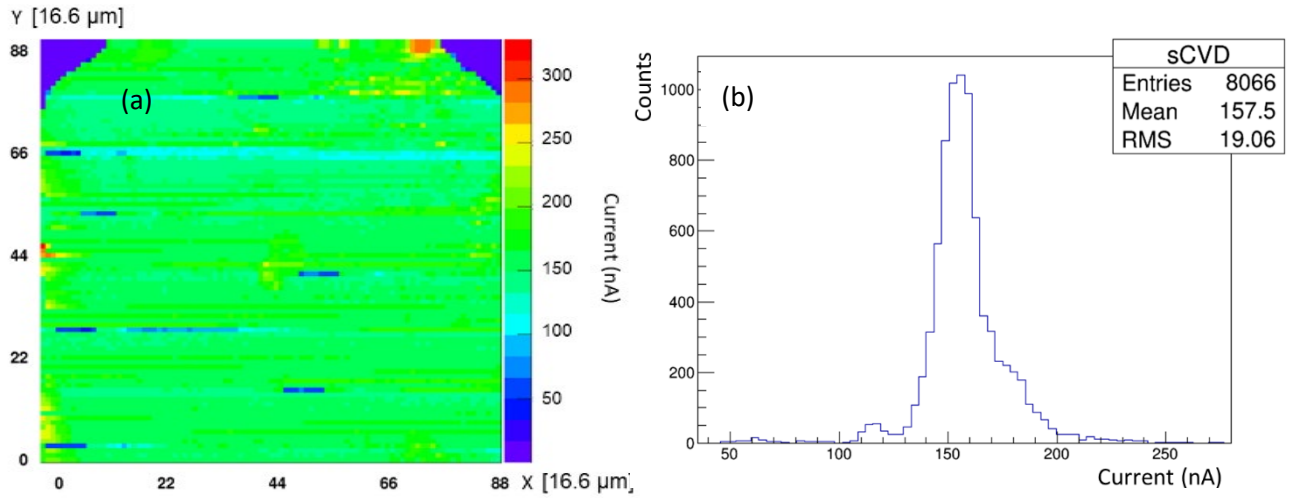


Figure 7 Histogram of the measured current (a) on the surface map (b) of a  $0.45 \times 0.45 \text{ cm}^2 \times 518 \text{ }\mu\text{m}$  sCVD diamond detector from Element 6.

293

294 In Figure 7 a current map is shown for a  $0.45 \times 0.45 \text{ cm}^2 \times 518 \text{ }\mu\text{m}$  sCVD diamond detector from  
 295 Element 6. The current was not measured with the Keithley electrometer but signal pulse waveforms  
 296 were recorded with the DSO from Lecroy and current values were deduced from off-line analysis (using  
 297 pulse integration, see section 2.6). The diamond was biased at +500 V, the whole diamond surface was  
 298 characterized with a scan position stepping size of  $16.6 \text{ }\mu\text{m}$ , and the signal integration time at each point  
 299 was set to 1 s. Periodic horizontal lines can be observed that arise from the beam refill every 60 minutes  
 300 in this particular bunch mode configuration. These beam variations affect the results. The dark blue  
 301 segments are correlated with the beam refill. The sudden beam increase process leads to an injection of  
 302 charges. At first it was observed that the diamond signal decreased strongly (dark blue segment) before  
 303 going up (yellow segment). It should be mentioned here that the 2D map is filled from left to right at  
 304 each row. Charge may certainly saturate traps inducing trap priming which leads then to an overshoot  
 305 due to the space charge build up, as described in [42]. Anyway, the diamond response is very  
 306 homogeneous if we exclude a singular point at the top right corner on Figure 7 (a) which is located near  
 307 the circular collection electrode. This suggest a beam-induced charge injection from a defective surface  
 308 site. Similar results were obtained [36] at ESRF with electronic grade sCVD samples which were  
 309 patterned with Ni-TiC contacts: a uniform spatial response - flat within 0.2% - was seen when the  
 310 devices were mapped with a sub-micron synchrotron collimated X-ray beam. Furthermore, X-ray beam  
 311 absorption occurring in the Ti metal layers was also quantified. As previously discussed in section 2.1,  
 312 this study, conducted by some of the authors of present paper, guided our choice towards the use of  
 313 aluminum contact rather than titanium based ones.”

314 Assuming that the integral of the current over each pulse must be equal to the total charge  $Q_{\text{beam}}$  created:

315

316 
$$Q_{\text{beam}} = \frac{\Phi_{32\text{mA(ph)}} \times T_{\text{beam}} \times E_{\text{ph}} \times R_{\text{diamond}} \times e}{E_{\text{eh/diamond}}}, \quad (4)$$

317 where  $\Phi_{32mA}(ph)$  has been calculated before and was found equal to  $1.79 \cdot 10^9$  ph/s,  $E_{ph}$  and  $T_{beam}$  have the  
 318 same definitions as those used in equation (1).  $E_{eh/diamond}$  is the required energy in diamond to create  
 319 electron hole pairs (13.6 eV [43] [44]). Finally,  $R_{diamond}$  is the fraction of the beam energy deposited  
 320 in the diamond thickness, which can be calculated as follows:

$$321 \quad R_{diamond} = 1 - \frac{I_{ph-out}}{I_{ph-in}} = (1 - e^{-\mu\rho d}), \quad (5)$$

322 where:

- 323 •  $\mu$  is the mass absorption coefficient (for 8.5keV photons in carbon ,  $\mu=4.025 \text{ cm}^2\text{g}^{-1}$ ),
- 324 •  $\rho$  is the mass density ( $3.517 \text{ g cm}^{-3}$ ),
- 325 •  $d$  is the diamond sample thickness ( $518 \text{ }\mu\text{m}$ ).

326 Using the known mass density of single crystal diamond of  $3.517 \text{ g cm}^{-3}$ , equation (5) leads to an energy  
 327 deposition per individual X-ray beam pulse in the  $518 \text{ }\mu\text{m}$  thick sCVD diamond:  $E_{diamond} = 5.2 \text{ MeV}$ .  
 328 This implies that  $Q_{beam} = 61 \text{ fC}$  and  $I_{mean} = Q_{beam}/T_{beam} = 86 \text{ nA}$ .

329 The difference between calculation and measurement can be explained by the fact that the gain of the  
 330 CIVIDEC C2 used is not precisely known, while in equation (3) a nominal value of 40 dB was used to  
 331 calibrate the map in Figure 7. The calculated value 86 nA would correspond to a real amplifier gain of  
 332 45 dB. This analysis also assumes complete charge collection within a sCVD diamond, which has been  
 333 confirmed in previous laboratory measurements we performed with an  $^{241}\text{Am}$  5.5 MeV alpha source test  
 334 bench [37].

335

### 336 3.2 Polycrystalline diamond (pCVD)

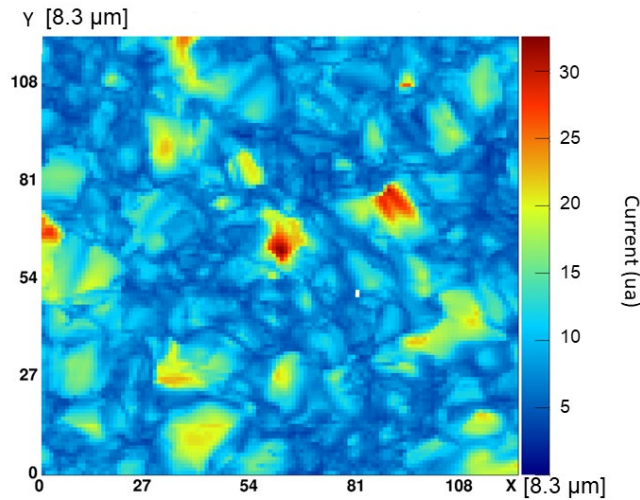


Figure 8 Surface map ( $1 \text{ mm}^2$ ) of a  $1 \times 1 \text{ cm}^2 \times 500 \text{ }\mu\text{m}$  pCVD from Element 6.

337

338 Surface mapping of several detectors was first performed using the current integration mode. Figure 8  
 339 shows a map from a disk metallized region of a polycrystalline detector (total diamond size  $1 \times 1 \text{ cm}^2 \times$   
 340  $500 \text{ }\mu\text{m}$ ), pCVD from Element 6. The diamond was biased at  $-500 \text{ V}$ . A piece of the diamond surface  
 341 ( $1 \text{ mm}^2$ ) was characterized with a position step size of  $8.3 \text{ }\mu\text{m}$  and the signal integration time at each  
 342 point was set to 200 ms. The colour scale corresponds to the charge collection efficiency measured by  
 343 the Keithley electrometer whose gain was set in auto range. Consequently, the vertical scale is displayed  
 344 in arbitrary units. In fact, the case presented here corresponds to the worst signal response variations

345 measured, chosen to show good contrast in the image. Clearly, the response of the detector reflects the  
 346 spatial distribution of grain boundaries in the polycrystalline material under the micro-focussed X-ray  
 347 beam. In this sample a factor 6 of difference is observed between the highest signal response (some few  
 348 hot spots scattered on the whole surface) and the lowest signal response areas. Such results confirmed  
 349 the previous analysis reported in [45] which was obtained on the same beamline at ESRF in 2002. The  
 350 sensitivity maps of a 300- $\mu\text{m}$ -thick commercially available pCVD detector grade diamond, metallized  
 351 with gold contacts and exhibiting an average 80–100  $\mu\text{m}$  grain size range on both sides, were shown in  
 352 [45], and the authors concluded that current measurement exhibited very strong variations that were  
 353 associated with the grain boundaries within the material.  
 354

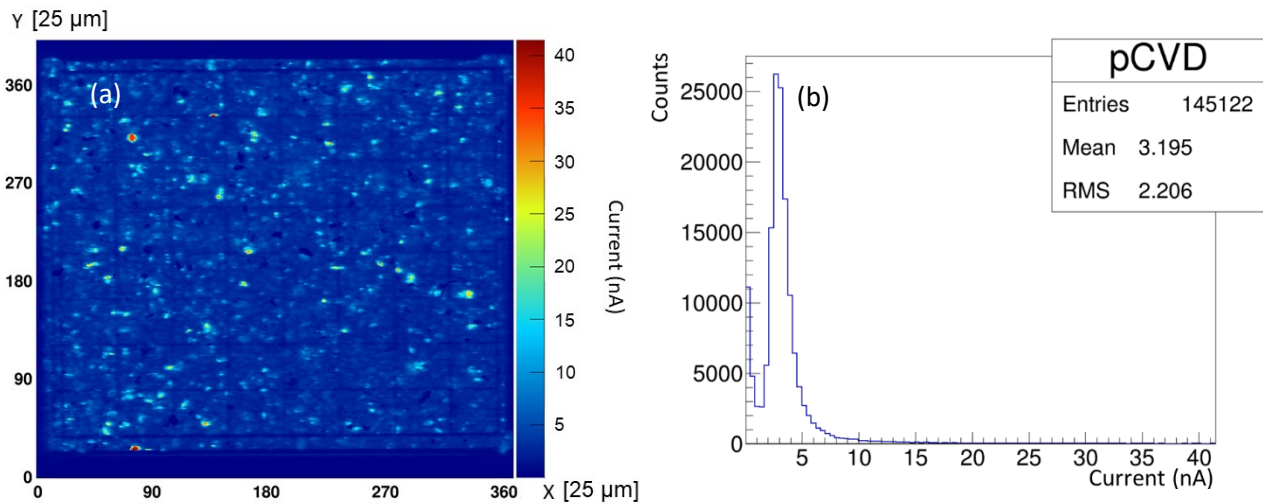


Figure 9 Histogram of the measured current normalized to  $\Phi_{32\text{mA}}(\text{ph}) = 1.79 \cdot 10^9 \text{ ph/s}$  (b) on the surface map (a) [32] of a double side striped  $1 \times 1 \text{ cm}^2 \times 300 \mu\text{m}$  pCVD diamond detector from Element 6

355

356

357 Figure 9 shows the striped pCVD detector map ( $1 \times 1 \text{ cm}^2 \times 300 \mu\text{m}$  detector from Element 6) calibrated  
 358 according to the method described in section 2.5. The whole diamond surface was characterized with a  
 359 position step size of 25  $\mu\text{m}$  and the signal integration time at each point was set to 100 ms. The diamond  
 360 was biased at +300 V. The non-uniformity of the diamond response as indicated by the colour scale on  
 361 the right reflects some hot spots (as observed in Figure 8) distributed homogeneously over the surface  
 362 and demonstrates the grain boundary distribution in the diamond material, itself resulting from the  
 363 pCVD diamond growth [46]. The authors of [47] observed in similar conditions under micro X-ray beam  
 364 radiation, evidence of localised space charge build-up mechanisms in CVD diamond, and demonstrated  
 365 that the observed hot-spots could be related to the accumulating of charges at defect sites. The presence  
 366 of these spots did not, however, greatly affect the homogeneity of the detector response. Indeed, the  
 367 histogram of the current values measured on the entire surface (including the non-metallised areas  
 368 between the contact strips) exhibit a single peak at  $\sim 3 \text{ nA}$ , with 2 nA RMS dispersion. Given the  
 369 theoretical calculation presented in equation (4), for a 300  $\mu\text{m}$  thick diamond we would expect  $R_{\text{diamond}} = 0.35$ .  
 370 Assuming a charge collection efficiency of 100% this results in  $I_{\text{mean}} \sim 58 \text{ nA}$ . The experimental  
 371 results obtained imply that the spatially averaged charge collection efficiency is only 5.5 %, but Figure  
 372 9 exhibits a homogenous distribution of “hot spots” in which the mean current reaches up to 40 nA,  
 373 corresponding to 70 % charge collection efficiency.

374

375 **3.3 Diamond growth on iridium (DOI).**

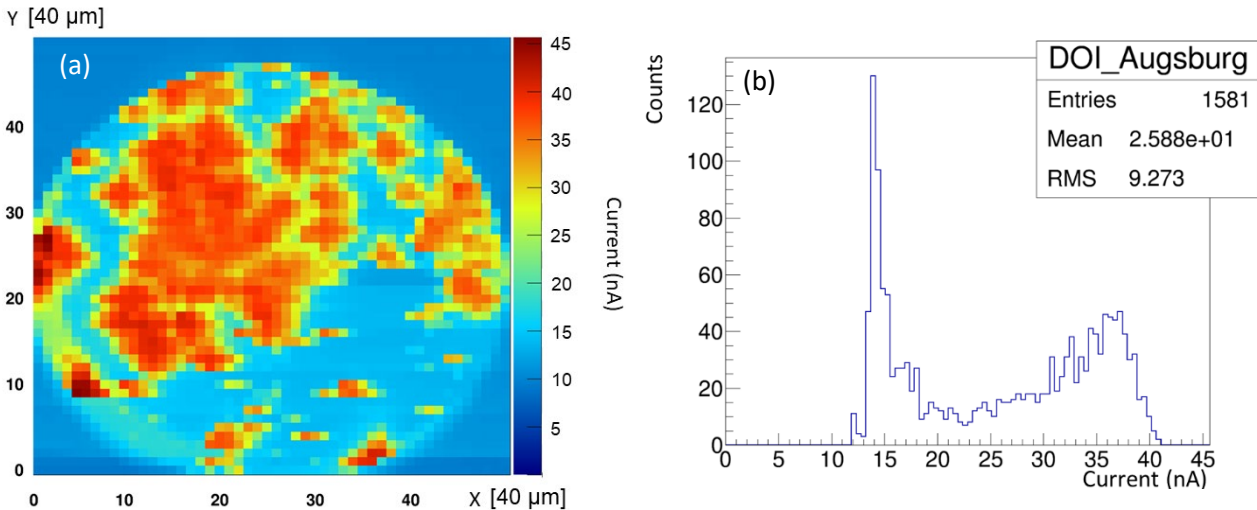


Figure 10 Histogram of the measured current normalized to  $\Phi_{32mA}(ph) = 1.79 \cdot 10^9 \text{ ph/s}$  (b) on the surface map (a) of a  $0.5 \times 0.5 \text{ cm}^2 \times 300 \text{ }\mu\text{m}$  DOI detector from Augsburg University, biased at -300V.

376 Figure 10 shows a current map done with a disk-metallized  $0.5 \times 0.5 \text{ cm}^2 \times 300 \text{ }\mu\text{m}$  DOI detector from  
 377 University of Augsburg, biased at -300 V. The whole diamond surface was characterized with a position  
 378 step scan of  $40 \text{ }\mu\text{m}$ , and the data acquisition time on each point was 200 ms. The signal response is  
 379 clearly inhomogeneous, as illustrated by the colour map and the histogram of the current distribution  
 380 obtained for the entire disk-metallized diamond surface. The theoretical response value is at the level of  
 381 58 nA, estimated for a constant photon flux of  $\Phi_{32mA}(ph) = 1.79 \cdot 10^9 \text{ ph/s}$ , a diamond thickness of 300  
 382  $\mu\text{m}$ , and a charge collection efficiency of 100%. In Figure 10 (b) two histogram maxima are observed:  
 383 a narrow one at  $\sim 14 \text{ nA}$  corresponding to the low response zones (blue regions on the map), and a  
 384 broader one at  $\sim 37 \text{ nA}$  with a long tail towards lower current intensities. It should be mentioned that  
 385 previous analyses [31] have demonstrated that this type of diamond has good time and energy resolution  
 386 for measurements that are averaged over the whole diamond surface. The mean value of the distribution  
 387 on Figure 10 (b) corresponds to a charge collection efficiency of 45 %. In areas coloured red in the  
 388 response map the charge collection reaches up to 71 %. However, the detector exhibits extensive areas  
 389 with very reduced current intensity and low collection efficiency (8%) that indicate charge carrier  
 390 trapping at dislocation sites [48]. In the present experiment, the signal is produced by both electron and  
 391 hole drift within the sample, so it is impossible to distinguish between electron or hole trapping.  
 392 However, in reference [49], the transport of both carriers is shown to be affected by dislocations.

393 In Figure 11 and Figure 12 two surface maps of two different striped DOI diamond manufactured by  
 394 Audiatic (a spin-off company of the University of Augsburg) of bigger size ( $1 \times 1 \text{ cm}^2$ ) are shown,  
 395 together with their corresponding X-ray beam transmission maps.

396 In Figure 11 (a) the DOI-1 diamond was biased at +300 V. The whole diamond surface was measured  
 397 with a position step of  $20 \text{ }\mu\text{m}$  and data acquisition time on each point of 200 ms. In Figure 11 (b) and  
 398 (c), the diamond was biased at -300 V and measured with a position step of  $50 \text{ }\mu\text{m}$ , and data acquisition  
 399 time on each point of 100 ms.

400



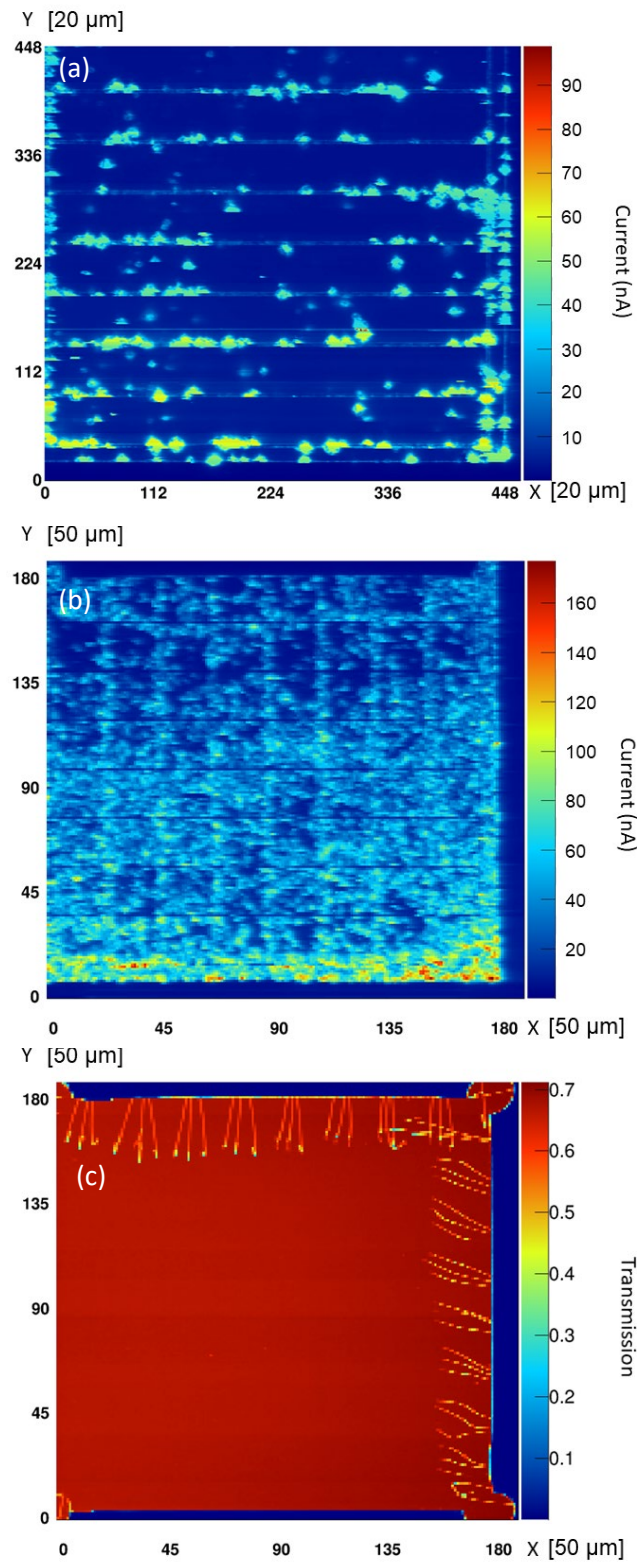


Figure 11 Surface maps of the  $1 \times 1 \text{ cm}^2 \times 300 \text{ } \mu\text{m}$  DOI-1 detector from Audiatec, (a) biased at +300 V (b) biased at -300 V, (c) shows the X-ray beam transmission map in which the location of the wire bonds are clearly observed. Results are normalized to  $\Phi_{32\text{mA}}(\text{ph}) = 1.79 \cdot 10^9 \text{ ph/s}$ .

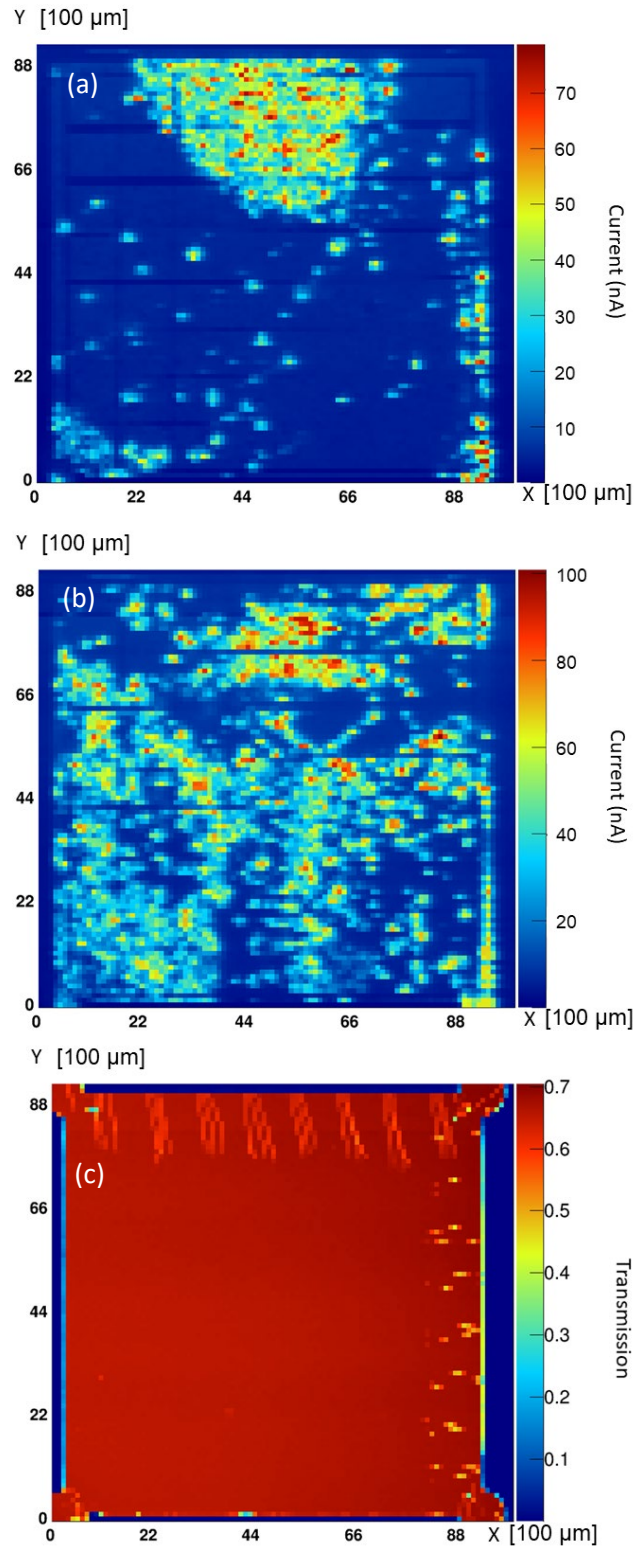


Figure 12 Same as Figure 11, with the  $1 \times 1 \text{ cm}^2 \times 300 \text{ μm}$  DOI-2 detector (manufactured by Audiatic) biased at +300 V (a), -300V (b) and the transmission map (c). Results are normalized to  $\Phi_{32\text{mA}}(\text{ph}) = 1.79 \cdot 10^9 \text{ ph/s}$ .

403 In Figure 12 (a), the DOI-2 diamond with non-regular strip spacing (see Figure 5 (b)), was biased at  
404 +300 V and in Figure 12 (b) at -300 V. The whole diamond surface was characterized in both cases with  
405 a position step of 100  $\mu\text{m}$  and a data acquisition time on each point of 200 ms.

406 In both Figure 11 (a) and (b) and Figure 12 (a) and (b) we observe that the DOI calibrated maps show  
407 inhomogeneous current distribution over the diamond surface, as also shown in Figure 10. For each DOI  
408 sample, the (a) and (b) maps were obtained using the same magnitude of diamond bias voltage but  
409 applied with opposite sign. The comparison of DOI-1 (a) versus (b) and DOI-2 (a) versus (b), reveals  
410 obvious discrepancies in the current distributions. A complete review of the results obtained with DOI  
411 diamond samples is given in [50], where the authors demonstrated that in DOI, the transport of the holes  
412 is efficient whereas electron transport is poor. This obviously corroborates our present results. Indeed,  
413 either the horizontal or vertical electrodes show higher contrast in Figure 11 (a) and (b) and Figure 12  
414 (a) and (b), depending on the chosen bias, as we would expect if a single type of carrier contributes to  
415 the observed signal. If the two charge carriers were collected with equal efficiencies, then on the same  
416 map we would not be able to distinguish between the two types of electrodes.

417 The alignment of hot spots along the right vertical edge of the detectors, Figure 11 and Figure 12,  
418 corresponds to the presence of wire bonds which caused an enhancement of the XBIC signal, as already  
419 observed in [48]. The transmission maps, Figure 11 (c) and Figure 12 (c), obviously show this same  
420 contrast due to X-ray absorption in the Al wire bonds, which results in the production at the bond  
421 interface of more hot, ionizing electrons. However, the higher signals observed were also likely to result  
422 from the 'damage' at the diamond-metal interface resulting from the mechanical impact of the Al wedge  
423 wire bonding process. As pointed out above, on Figure 11 (a) and (b), the shape of the electrodes can be  
424 clearly distinguished. The darker areas reflect a defect in the collection of charges in the inter-strip  
425 region. This can be seen very clearly in the case of DOI-2 (Figure 12 (a) and (b)) where the size of this  
426 inter strip spacing varies from 50  $\mu\text{m}$  to 200  $\mu\text{m}$ . Indeed, it was possible to demonstrate by simulation  
427 (COMSOL Multiphysics [51]) that the electric field decreases between two adjacent electrodes which  
428 results in a significant decrease in the observed current. This is also visible in Figure 9 for the pCVD  
429 diamond. This implies that if we want to improve the charge collection, it will be necessary to reduce  
430 the inter strip width (see section 4). The significant increase in the signal at each electrode edge reflects  
431 a local increase of the electric field. The consequence of reversing the polarity is that the same type of  
432 carrier will no longer be collected by a vertical electrode (for example), but instead by a horizontal  
433 electrode. In the case of DOI diamonds, if we assume that only the hole charge carriers are collected  
434 with high efficiency, then the observation that the horizontal and vertical electrodes do not show equal  
435 contrast could reflect the "blocking", Schottky-like nature of the aluminum contacts. We note that the  
436 observed differences in diamond signal response for positive or negative high voltage biasing of the  
437 sample is not specific to the DOI samples, since it was also obvious in the other samples. Indeed, leakage  
438 currents show an asymmetry in Figure 2. Furthermore, after we obtained the present XBIC results, tests  
439 using EBIC (Electron Beam Induced Current) carried out at the Institut Néel, Grenoble, on samples of  
440 monocrystalline diamonds suggest that the choice of aluminum for the electrodes results in Schottky-  
441 like contacts of varying quality which likely explain the asymmetry. A study on improving contacts is  
442 underway.

443 For both DOI-1 and DOI-2 diamonds biased at -300 V, the histograms of the  $I_{\text{mean}}$  current over the  
444 metalized diamond surface are displayed in Figure 13. As before, the theoretical signal value is 58 nA,  
445 estimated assuming a constant photon flux of  $\Phi_{32\text{mA}}(\text{ph}) = 1.79 \cdot 10^9$  ph/s, a diamond thickness of 300  $\mu\text{m}$   
446 and a charge collection efficiency of 100%. DOI-1 shows a broad peak (similar to that of Figure 10 (b))  
447 whereas DOI-2 shows a narrower single peak with a long tail spreading to higher current values. In  
448 Figure 13 (a), the mean value is 34.16 nA, corresponding to a charge collection efficiency of 60 %,  
449 whereas in Figure 13 (b) it reaches only 40 %. Such analyses confirm the results already observed with  
450 the smaller disk-metallized diamond, laboratory measurement with alpha particles [37] and results from  
451 [50] and seem very representative of the varying area distribution of dislocation densities in diamond  
452 grown on iridium, unlike the previous pCVD sample tested in the same conditions.

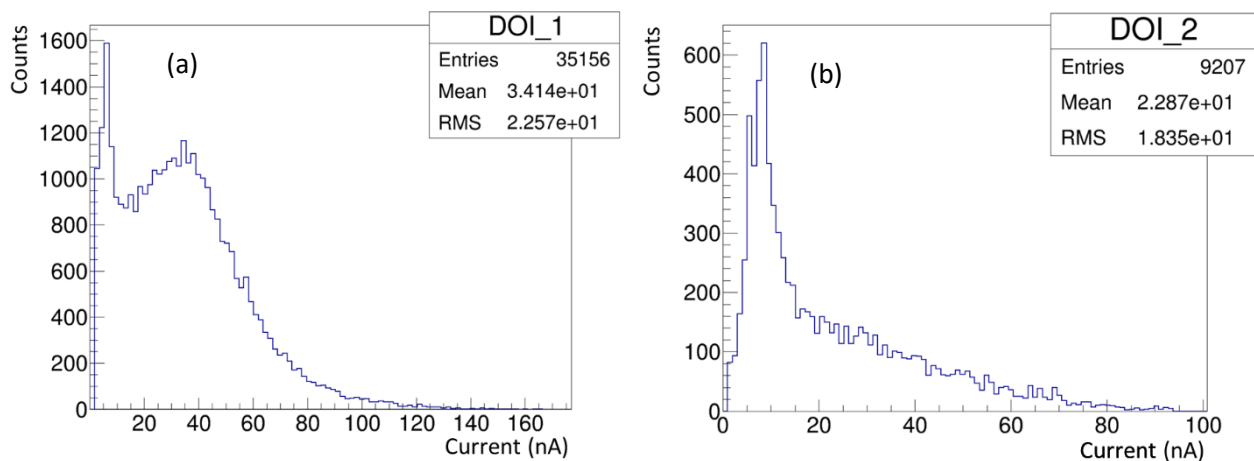


Figure 13 Histogram of the  $I_{\text{mean}}$  current over the diamond surface on the two different  $1 \times 1 \text{ cm}^2 \times 300 \mu\text{m}$  DOI samples (DOI-1 (a) and (b) DOI-2) biased at  $-300 \text{ V}$ . Results are normalized to  $\Phi_{32\text{mA}}(\text{ph}) = 1.79 \cdot 10^9 \text{ ph/s}$ .

454 **3.4 Compared performances and diamond selection to equip the first prototype of beam**  
 455 **hodoscope.**

456

457 On the one hand, the measured inhomogeneity of the striped DOI-1 and DOI-2 diamond current maps  
 458 at ESRF with  $8.5 \text{ keV}$  X-rays pulses was found to be prohibitive to envisage the readout of this diamond  
 459 type with striped electrodes as foreseen in the planned hodoscope design. Indeed, the signal amplitude  
 460 will depend on the location where the particle interacts in the DOI. In the best cases we reached 70% of  
 461 charge collection efficiency. Depending on the type of particle and its energy that we want to detect,  
 462 this may lead to varying detection efficiency.

463 On the other hand, the sCVD diamond exhibited excellent performance but the available size for ‘off  
 464 the shelf’ detectors are still very small. In order to cover close to  $1 \text{ cm}^2$  area four  $0.45 \times 0.45 \text{ cm}^2$  sCVD  
 465 diamond are necessary to be assembled in a mosaic arrangement.

466 Such an assembly is to be considered in the future but at first it was decided to choose the  $1 \times 1 \text{ cm}^2$   
 467 pCVD detector to equip the first hodoscope prototype and to evaluate its performances in terms of  
 468 detection efficiency and time resolution under  $8.5 \text{ keV}$  X-ray pulsed beams.

469

470 **4. Performances of the pCVD diamond based first hodoscope prototype in pulse mode.**

471

472 In this part of the work our objective was to select X and Y strips and to scan them with the micro  
473 focused beam. We aimed to evaluate, at each micro-beam location, two parameters: the striped pCVD  
474 detector X-ray detection efficiency and time resolution. By detection efficiency, we mean the capability  
475 of the detector to act as a trigger on a measurable amplitude signal, using a fixed threshold above  
476 background level. Our goal was to study the impact of the presence of the observed inhomogeneities on  
477 these two parameters which would affect in-beam performance of the future diamond hodoscope we  
478 plan to build.

479

480 **4.1 X-ray detection efficiency measured over various top-bottom strips crossing at micro-beam**  
481 **positions.**

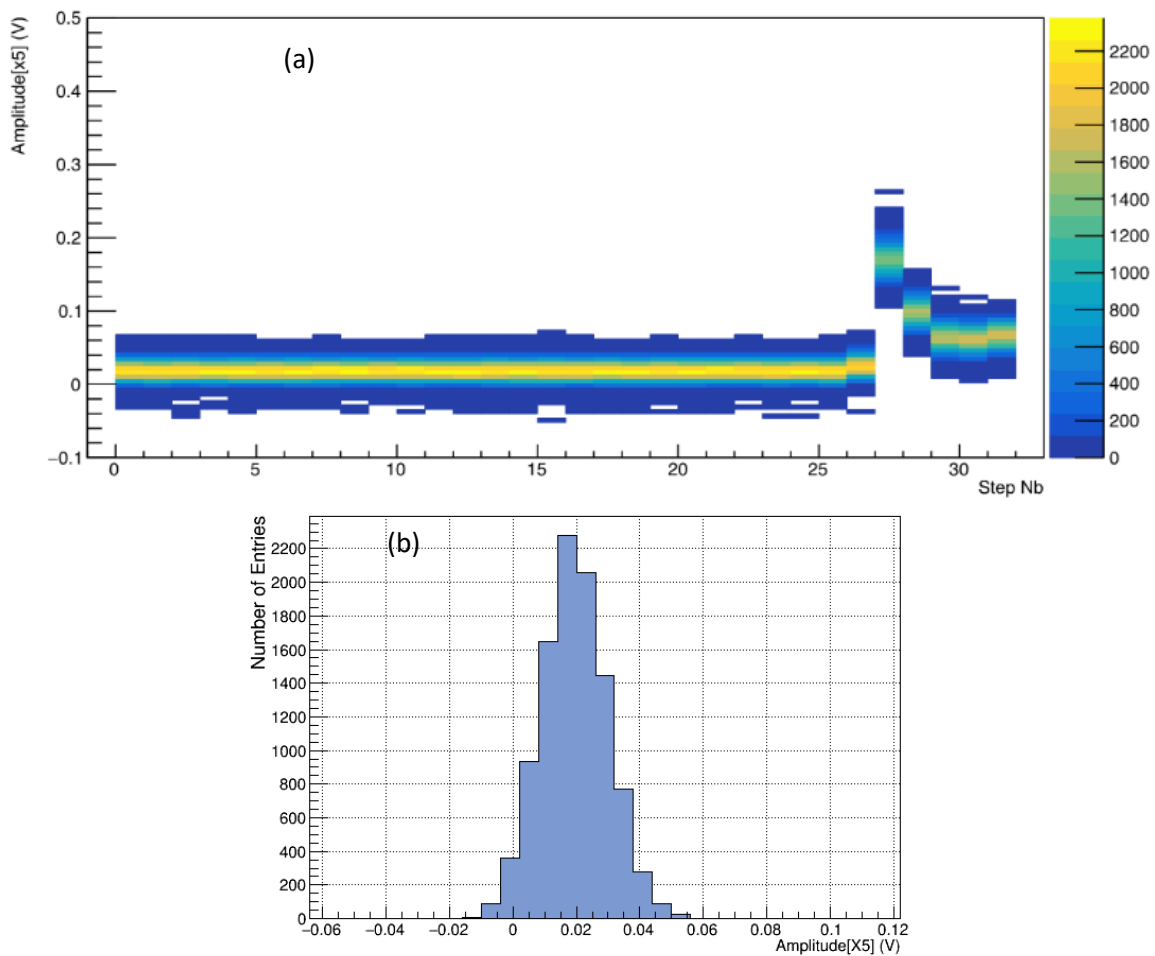


Figure 14 The maximum amplitude on strip X5 displayed for all irradiation positions labelled in step number (a), and the corresponding Y projection at step number 17 (b).

482

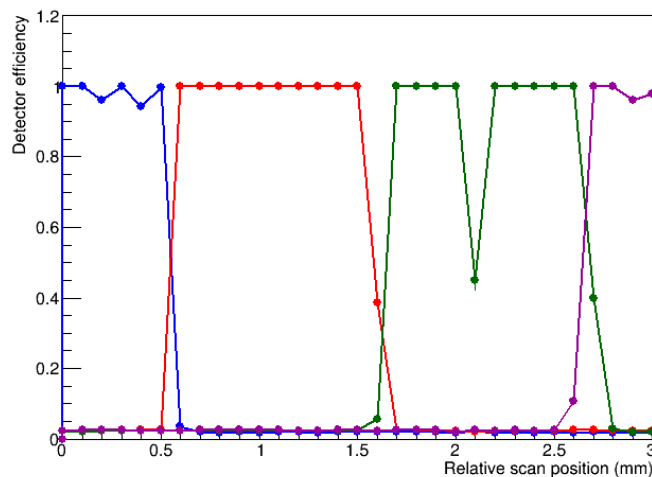


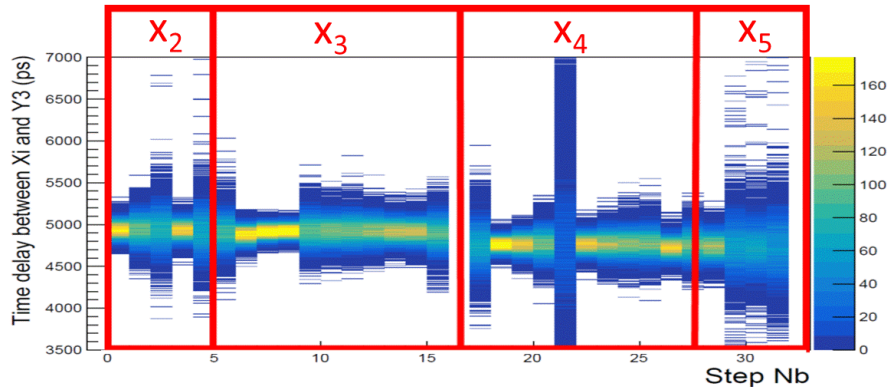
Figure 15 X-ray detection efficiency measured with the striped  $1 \times 1 \text{ cm}^2 \times 300 \mu\text{m}$  pCVD diamond from Element 6 (step size  $100 \mu\text{m}$ , interstrip size  $100 \mu\text{m}$ ).

483 To carry out this analysis, 4 strips in X (namely X2, X3, X4, X5) and 1 strip in Y of the prototype  
 484 hodoscope were connected to the Wavcatcher data acquisition system (see Figure 3 (c)). As previously  
 485 explained, an external timing trigger was provided by the synchrotron storage ring radio frequency. A  
 486 horizontal scan on the 4 strips in X was done with  $100 \mu\text{m}$  position steps with signal responses acquired  
 487 for  $10^5$  X-ray pulses at each position step. It should also be noted that, as the scan step is equal to the  
 488 size of the inter strip, at most we expect to have a single point of measurement at this location. The  
 489 detector was biased at  $-500 \text{ V}$  ( $1.6 \text{ V}/\mu\text{m}$ ) to further improve signal charge collection. Figure 14 (a)  
 490 shows the amplitude distribution of strip X5, for 32 different irradiation positions (Step Number). When  
 491 X5 was not irradiated, i.e. from step numbers 0 to 26, the amplitude corresponds to electronic noise. The  
 492 diamond noise response is rather uniform for the different irradiation positions. In Figure 14 (b), the  
 493 histogram of amplitudes registered at the step number 17 is shown as an example. The efficiency has  
 494 been calculated for each strip and for each incident position as the ratio between the number of detected  
 495 X-ray pulses per number of trigger events. The number of detected X-ray pulses corresponded to the  
 496 number of signals above a fixed threshold of  $40 \text{ mV}$ , which, according to Figure 14, was the optimum  
 497 level to avoid excessive triggering on electronic noise. Results are shown in Figure 15 as a function of  
 498 the X-axis hit coordinate. The various colours correspond to efficiencies for adjacent strips of the  
 499 diamond detectors.

500 As observed in Figure 15, the strip detection efficiency is very close to 100% as evaluated over four  
 501 adjacent strip crossings, except in the strip gaps: for one gap position ( $x=0.6 \text{ mm}$ ), the efficiency is 1,  
 502 for the two others it falls to 30-35% ( $x=1.7 \text{ mm}$ ,  $x=2.8 \text{ mm}$ ) on only one of the two adjacent strips  
 503 involved in charge sharing (no cross talk between adjacent strips). This result motivates us to decrease  
 504 the inter strip gap for the next version of the prototype. The point at position =  $2.2 \text{ mm}$  with 40 % may  
 505 correspond to a very localized defect on the strip. We can certainly link this local result to the X-BIC  
 506 2D maps made on the same detector. The current integration response map in Figure 9 shows the relative  
 507 heterogeneity of the response of the detector (and therefore of its charge collection capacities) as a  
 508 function of the part of volume irradiated. It is possible that this lack of detection efficiency is due to  
 509 irradiation of an area of lower response on the detector surface. Apart from this singular point, the  
 510 response of this detector remains relatively homogeneous as far as fixed threshold discrimination is  
 511 used.

512

513 **4.2 Time resolution measured over various top-bottom strips crossing at different micro-beam**  
 514 **positions.**



**Figure 16** Time distribution over each micro-beam position on Xi-Y3 strip crossing (materialized by the red lines) with i ranging from 2 to 5.

515

516 In this experiment, the selected Y strip remains the same (Y3) but the X strips vary over the surface  
 517 detector as described in the previous section (from X2 up to X5). The waveforms were recorded as  
 518 before for the X-ray detection efficiency measurement. In total,  $10^5$  beam pulses were recorded at each  
 519 step corresponding to the micro-beam position over the considered strips crossing. To obtain the time  
 520 resolution, a numerical Constant Fraction Discrimination (CFD) was used by averaging the background  
 521 on each waveform for the calculation of the baseline, determining the maximum height of the pulses  
 522 and interpolating the 50 % rise time value (Figure 16). The distribution of the time difference measured  
 523 simultaneously for the two faces of a detector was characteristic of the time jitter of the readout chain  
 524 connected to the detector. Figure 17 (b) illustrates this jitter measured on step strip crossing number 14  
 525 located on X3 (average of  $10^5$  points). This point was chosen as a representative average, with a standard  
 526 deviation of 108 ps deduced from a Gaussian fit. Figure 17 (a), shows the time resolution 65 ps RMS  
 527 measured on one of the best cases, step number 8.

528 The correlation between the average signal amplitude and the time resolution (each point in the graph is  
 529 the average of  $10^5$  recorded X-ray pulses, as for strip crossing number 14 in Figure 17) is shown in  
 530 Figure 18. The X-axis error bars indicate the RMS of the signal amplitude distribution as measured at

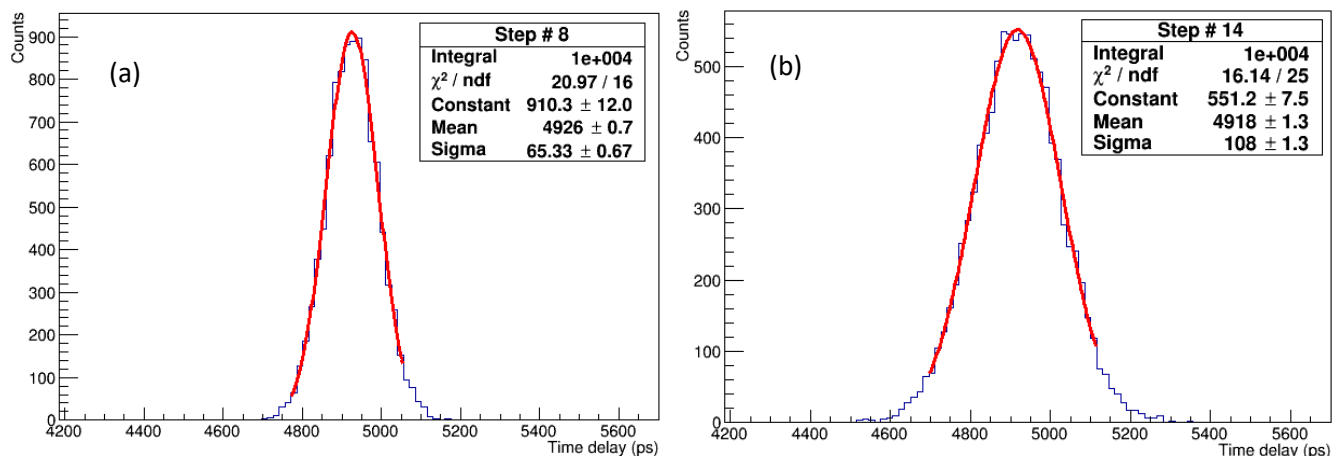


Figure 17 Time resolution (Gaussian fit) measured on step number 8 (strip crossing X3 – Y5) (a) and on step number 14 (b) for changing micro-beam positions on the pCVD detector.

531 each micro-beam position step number. The error in time resolution is the uncertainty on the fit relative  
 532 to the standard deviation parameter. In the present experiment, the deposited energy is constant and is  
 533 linked to the diamond thickness but the charge collection efficiency varies from one point to another on  
 534 the diamond surface (XBIC experiment) resulting in a variation of the signal amplitude. The adjustment  
 535 was made by a function proportional to the inverse of the maximum amplitude of the signal. It can be  
 536 clearly observed that the data are well fitted by such a function. It implies that the collected energy is  
 537 proportional to the signal amplitude, as expected, and, in addition, that the recorded signal pulses are  
 538 not degraded at beam positions where the diamond response is lower.

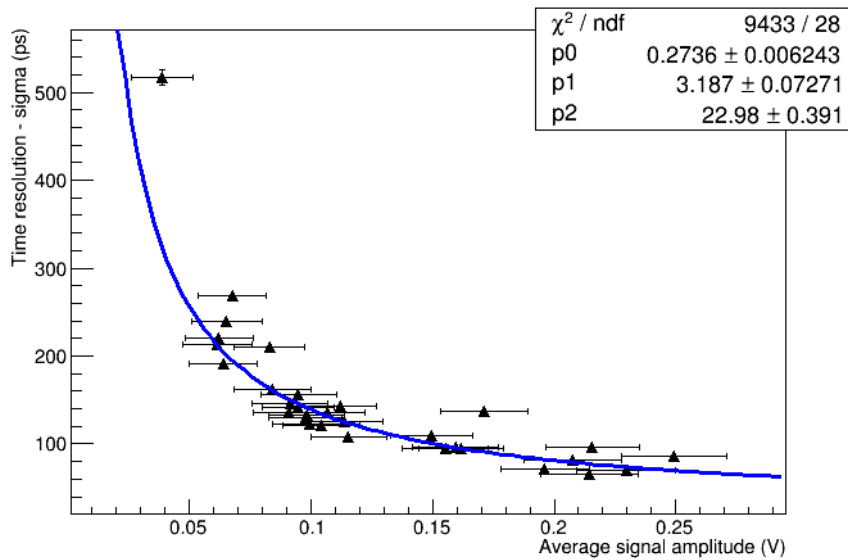


Figure 18 Correlation between the average signal amplitude and the time resolution for beam position over Xi-Y3 with  $i$  ranging from 2 to 5 (each point is the average of  $10^5$  X-ray pulses). Error bars in the Y direction are within the point size.

539 Figure 19 shows the observed time resolution ( $\sigma = \text{RMS}$ ) as a function of step number (micro beam  
 540 position). The error shown on the y-axis is the error on the fit for the  $\sigma$  parameter. The scatter of  
 541 results correspond to a time resolution ranging from 60 ps to 260 ps if we exclude the singular point  
 542 already observed in previous plots. Although the distribution among the various tested points is large,  
 543 the barycentre of the point cloud is  $150 \pm 87$  ps, a value we are specifically seeking to achieve with the  
 544 future beam hodoscope. It should be noted that in this readout system, in particular the preamplifiers,  
 545 has not yet been optimized, and we are confident that a timing resolution below 100 ps can be obtained  
 546 for carbon ion beams. In the present case, the energy deposit per pulse is 3.4 MeV at maximum (just  
 547 after the storage ring refill). For proton beams, where the energy deposit per single proton is below 1  
 548 MeV, such a setup will be able to detect bunches of several tens of protons with the desired efficiency  
 549 and time resolution.

550 In comparison, the time resolution between the two faces of the disk metallized sCVD diamond studied  
 551 in paragraph 3.1 was found to range between 25.1 ps (CIVIDEC C2) and 53.8 ps (LPSC), i.e. depending  
 552 on the type of current-voltage preamplifier used (more details are given in reference [32]). There is about  
 553 a factor 2 of difference that relies on the readout electronic fast preamplifier. Furthermore, the  $1 \times 1 \text{ cm}^2$   
 554  $\times 500 \mu\text{m}$  pCVD results shown in Figure 8, with disk-metallization and higher detector thickness (500  
 555  $\mu\text{m}$  instead of 300  $\mu\text{m}$ ) exhibited a time resolution of  $\sim 72$  ps using readout by two CIVIDEC C2  
 556 preamplifiers, as reported in reference [32]. So we can conclude that all these results are consistent and  
 557 that a means to further improve this resolution will be to improve our fast front-end readout electronics.



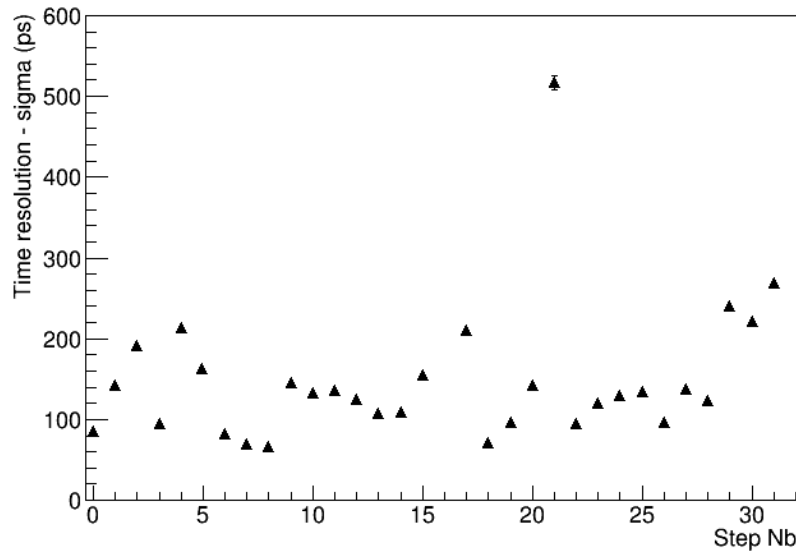


Figure 19 Time resolution versus Step number which corresponds to the beam position over Xi-Y3 with  $i$  ranging from 2 to 5 (each point is the average of  $10^5$  points). Error bars are within the point size.

559

560 **5. Summary and discussion on measured performances in XBIC in the perspective of the future**  
 561 **beam tagging hodoscope development for hadrontherapy monitoring.**

562

563 In the first part of the XBIC measurements, the three types of diamond samples sCVD, pCVD and DOI  
 564 were tested in current mode. This enabled an evaluation of their performance in terms of charge  
 565 collection efficiency over their entire surfaces, as probed by a micron size beam probe measuring every  
 566 few tens of microns. Laboratory experiments on an alpha particle test bench had already made it possible  
 567 to demonstrate that the charge collection efficiency of the sCVD diamond was 100% while that of pCVD  
 568 gave an area average of 30% and that of DOI 40% [37]. However, these experiments were made with a  
 569 radioactive source, so that the impact of the alpha particles on the surface of the diamond are at low  
 570 rates and random in time and spatial position. In consequence these measurements do not sufficiently  
 571 account for possible detector inhomogeneities. Therefore the measurements at the ESRF provided us  
 572 with additional knowledge on performance of diamonds as regards their spatial (non-) homogeneity.  
 573 This “homogeneity” parameter is a fundamental criterion for the hodoscope design involving a position  
 574 sensitive detector and whose development in terms of position calibration must not present any major  
 575 difficulty. In addition, the tunable intensity 8.5 keV X-ray pulses represented a maximum energy deposit  
 576 of 3.4 MeV and 5.2 MeV for diamond thicknesses of 300 and 500  $\mu\text{m}$ , respectively. This corresponds  
 577 to a collected charge of 40 and 61 fC (if we assume a collection efficiency of 100%), a similar energy  
 578 deposition that of a 400MeV/nucleon carbon ion beam in a 500  $\mu\text{m}$  thick diamond detector with an  
 579 average charge collection efficiency of 30%. Such a beam is very likely to be used in hadrontherapy.  
 580 The measurements carried out at the ID21 beamline are therefore completely transposable to one of the  
 581 future applications targeted by the hodoscope, and are a good indicator of its performance.

582 In a second part of the XBIC measurement, we made pulse measurements on the micrometer scale with  
 583 the pCVD demonstrator we selected thanks to the current map analysis. These measurements allowed

584 us to probe the detection efficiencies and to have time resolution estimates as a function of the point of  
585 impact of the ionizing beam on the strip crossings. Previous measurements carried out under conditions  
586 similar to those at the ESRF with disc metallized detectors coupled to three types of fast current - voltage  
587 preamplifiers (two from different manufacturers often cited in the literature for reading diamond signals,  
588 and one previously developed at LPSC used to readout the demonstrator discussed in this work) have  
589 enabled us to make comparisons [32]. If we look in detail at the time resolution distribution on the strip  
590 crossing, we can confirm that in certain points identified in the first part of the tests as "hot spots", the  
591 time resolution of the polycrystalline diamond approaches that measured with the single crystal  
592 diamonds in a previous experiment. We also conclude that for these measurements the performance of  
593 the electronic readout system is crucial and is an area requiring further work by us. Finally, at the strip  
594 crossings, the average time resolution is  $150 \pm 87$  ps RMS for an energy deposit of 3 MeV. As a  
595 consequence, we are confident of obtaining a timing resolution below 100 ps for carbon ion beams with  
596 a slightly improved version of the present electronics. With protons of 70 MeV, which is an energy used  
597 in certain clinical centers to treat eye tumors, the estimated energy deposit is 1 MeV, so it will be possible  
598 to detect bunches of several tens of protons with the desired efficiency and time resolution resolution  
599 (then the time resolution will be dominated by the bunch duration, not by the diamond).

600 All of these results motivate us to consider new developments in electronics. Since these experiments  
601 have been carried out, a new preamplifier design has been achieved in 2020 with improved performance.  
602 The gain is higher, the noise is comparable and the power consumption has been reduced more than 10-  
603 fold. This power reduction makes feasible the mounting of several chips in close proximity to the  
604 diamond strips leading to a higher-performance readout.

605 Furthermore, we are currently considering a solution consisting of an assembly of 4 monocrystalline  
606 diamonds of surface  $0.45 \times 0.45$  mm<sup>2</sup> (4 sCVD beam monitor) in order to better improve charge  
607 collection efficiency and thus to be able to meet the above specifications for the detection of single  
608 protons. The new experimental set-up under construction is intended for testing with 70 MeV protons  
609 at the ARRONAX facility in Nantes-France by the end of the year 2020. In addition, we have recently  
610 published first results on Prompt Gamma Timing with about 100 ps RMS Time of Flight resolution,  
611 using a fast monolithic scintillators and a single crystal diamond-detector beam trigger [52]. This result  
612 was obtained in ARRONAX with 70 MeV proton beam. Consequently, such a result makes us confident  
613 about the performance of the new 4 sCVD beam monitor version which we are currently developing.

## 614 **6. Conclusion**

615 Several diamond samples were tested under XBIC micro-beams. 2D maps have been drawn. Single  
616 crystal sCVD diamonds have shown a very homogeneous response. The pCVD diamonds show a  
617 scattered location of hot spots. However, the overall response of the detector is also very homogeneous.  
618 On the contrary, diamonds on iridium show very great inhomogeneity on the surface with large areas of  
619 defects.

620 The striped pCVD diamond was chosen to equip the first diamond based beam hodoscope. It was tested  
621 under X-rays. Results obtained are encouraging for the aimed development of a beam-tagging  
622 hodoscope with ToF capabilities. The various strip crossing tested showed time resolutions at the level  
623 of the targeted value of 100 ps RMS with excellent particle detection efficiency.

624 However, preserving an excellent ToF resolution at high proton energies seems challenging, particularly  
625 for pCVD. Another solution may consist in using either large area sCVD diamonds or a mosaic  
626 arrangement of sCVD. Large area sCVD are not commercially available at the moment but CVD  
627 diamond growth technologies progress quickly [53]. The next step of the development is then the  
628 assembling of four selected diamonds with optimized electronics for further characterization and tests  
629 under irradiation conditions close to clinical ones.

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631

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642

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