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IBIC CHARACTERIZATION OF AN ION-BEAM-MICROMACHINED MULTI-ELECTRODE DIAMOND DETECTOR

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ABSTRACT

Deep Ion Beam Lithography (DIBL) has been used for the direct writing of buried graphitic regions in monocrystalline diamond with micrometric resolution. As part of the development and the characterization of a fully ion-beam-micromachined solid-state ionization chamber, a device with interdigitated electrodes was fabricated by using a 1.8 MeV He⁺ ion microbeam, which scanned a 40 µm thick homoeptaxial detector grade diamond sample grown by chemical vapour deposition (CVD). In order to evaluate the ionizing-radiation-detection performance of the device, charge collection efficiency (CCE) maps were extracted from Ion Beam Induced Charge (IBIC) measurements carried out by probing different arrangements of buried micro-electrodes.

The analysis of the CCE maps allowed an exhaustive evaluation of the detector features, in particular the individuation of the different role played by electrons and holes in the formation of the induced charge pulses.

Finally, a comparison of the performances of the detector with buried graphitic electrodes with those relevant to conventional metallic surface electrodes evidenced the formation of a dead layer overlying the buried electrodes as a result of the fabrication process.

1. INTRODUCTION

In recent papers [1, 2] we described the fabrication of buried graphitic channels in monocrystalline diamond by Deep Ion Beam Lithography (DIBL). This technique consists of a selective damage of the crystal induced by MeV ion beams, which are focused down to a micrometer spot size and raster scanned on the sample along predefined linear patterns intersecting slowly-thinning metallic masks.
The damage induced by ions is localized mainly at their end of range, i.e. a few micrometers below the surface. The regions which experience a vacancy density overcoming a critical level, usually referred to as “graphitization threshold”, convert to a graphitic phase upon thermal annealing; elsewhere, the diamond structure is recovered.

With this method, highly conductive (resistivity of the order of 1 mΩ·cm) graphitic channels can be realized in single crystal diamond; their length is limited by the range of the micro-ion beam scanning system (typically several hundred micrometers); their minimum width is given by the beam spot size and their formation depth is defined by the nuclear stopping range of the ions in diamond (typically a few micrometers). Moreover, the presence of metallic masks, which modulate the nuclear stopping power of the ions, determines the emersion of the channels’ terminals at the surface, enabling the bonding of the graphitic channels to external electronic systems or the selective etching of the graphite for subsequent diamond micromachining processes [3].

The realization of highly conductive, optically opaque, chemically reactive graphitic channels embedded in a highly resistive, optically transparent and chemically inert diamond matrix is of potential interest in many sectors, e.g. for the realization of diamond 3D microstructures [4], microfluidic channels, innovative biosensors [5, 6], IR emitters [7] or bolometers [8]. An additional field of application is that of exploiting the DIBL to realize novel 3D architectures for ionizing radiation detection in order to increase their charge collection efficiency and to enhance their radiation hardness.

In order to characterize the carrier transport and recombination features of micromachined diamond detectors with buried electrodes, an analytical technique is needed to map the charge collection efficiency at a micrometer level of resolution, with a well defined probing depth profile suitable for analysis of the entire region where pulse signals are formed. In this paper we show that the Ion Beam Induced Charge [1, 9] technique fulfils these requirements. CCE maps obtained by raster scanning 4 MeV He⁺ ion micro-beams onto regions surrounding the buried graphitic electrodes provide valuable information not easily available otherwise on the electronic characteristics of the detector, such as the electric field distribution, the different role played by electrons and holes in the induced charge signal and the influence of the residual damage induced by the DIBL process to the diamond structure.

2. EXPERIMENTAL

The sample under test (sample 1) consisted of an intrinsic single-crystal ~40 µm thick homoepitaxial diamond layer grown on a commercial 4×4×0.4 mm³ high pressure high temperature (HPHT) Ib single crystal diamond substrate at the laboratories of Rome “Tor Vergata”
University, using a Microwave Plasma Enhanced Chemical Vapour Deposition (MWPECVD) process [10].

Graphitic channels buried into the diamond sample were made by DIBL [1, 2] at the AN2000 microbeam line of the INFN National Laboratories of Legnaro (I), using a 1.8 MeV He\(^+\) ion microbeam (~10 µm spot size) with an ion current of ~1 nA. The ion fluence was around 1.5·10\(^{17}\) cm\(^{-2}\). From a SRIM2011 simulation [11], these irradiation conditions are suitable for producing a vacancy density profile with a damage peak well above the graphitization threshold [12] and located at a depth of about 3 µm, as shown in Fig. 1.

The evaporation of variable thickness and slowly-thinning Cu masks onto the diamond surface before implantation ensured the electrical continuity of the buried channels with the surface, allowing for the modulation of the depth of the ion beam-induced damaged channels at their endpoints, as previously explained [1, 2].

After ion implantation, the Cu masks were removed from the surface. The sample was then annealed in vacuum at 1100 ºC for 2 hours, in order to convert the highly-damaged regions located at the ion end of range to a graphitic phase while removing the structural sub-threshold damage introduced in the layer overlying the above-mentioned damaged region (to which we will refer as the “cap-layer” in the following) [1, 2].

As a result of the process (see Fig. 2a), we fabricated four parallel buried graphitic channels in the sample, plus an additional channel orthogonal to them, each one being ~10 µm wide. The average spacing between channels was ~12 µm.

![Fig.1: Energy loss of 4 MeV He\(^+\) ions in diamond (left scale) and vacancy density profile in diamond generated by 1.47·10\(^{17}\) He\(^+\) ions of energy 1.8 MeV (right scale) as evaluated by SRIM 2011 simulation assuming a displacement energy value of 50 eV [2]. The horizontal line indicates the graphitization threshold (90 Vac nm\(^{-3}\) [12]).](image)
Fig.2: (a) Optical micrograph in transmission of sample 1. The graphitic channels buried in the diamond transparent matrix are visible as opaque stripes; at the corners the Cr/Al bonding pads connect the channels to the bias/amplification circuits schematically drawn in the figure. (b) Optical micrograph in transmission of sample 2 with the surface interdigitated Ti/Pt/Al electrodes and the relevant connections to the amplification/bias circuits.

To connect the graphitic channels to external electronic circuits, their emerging endpoints were contacted with 80 nm thick Cr/Al circular layers (150 µm diameter), which were used as bonding pads.

Electrical measurements between any pair of electrodes showed currents below the detection limit of our electrometer (<1 pA at ±100V applied bias), demonstrating both the high resistivity of the diamond matrix embedding the buried channels and a negligible surface leakage current.

Due to the epitaxial growth process and to the thickness of the HPHT substrate, the diamond sample was not equipped with a back electrode. Therefore, configuration and geometry of the electric field in the diamond bulk was entirely defined by the voltage applied at the buried electrodes.

In order to provide a comparison between buried graphitic channels and standard ohmic surface metallic contacts, a nominally identical intrinsic diamond sample (sample 2) was grown using the same CVD process. After CVD growth, Ti/Pt/Au (50/20/50 nm) finger contacts were patterned by a standard lift-off photo-lithographic technique and by thermal evaporation on the diamond surface. We performed a thermal annealing process at a temperature of 600 °C in Ar atmosphere in order to improve the ohmic interface between titanium and diamond [13].
A micrograph of the sample is reported in Fig. 2b, together with a sketch of the electrical connection arrangements adopted to perform the IBIC measurements. Also in this case, the current flowing between the two fingers was below our detection limit (i.e. < 1 pA at ±100 V).

IBIC measurements were carried out at the Ruđer Bošković Institute (RBI) microbeam facility using 4 MeV He$^+$ ions focused to a 4 µm diameter spot. We chose the energy of the ions to probe regions located beneath the graphitic electrodes, as results from the ionization profile shown in Fig. 1.

For the analysis carried out on sample 1, the microbeam raster scanned a rectangular area (120×150 µm$^2$) surrounding the buried electrodes. Ion current was low (<1000 ions·s$^{-1}$) in order to prevent damaging and to avoid pile-up effects. The electronic chain connected to the sensitive electrode consisted of a charge sensitive preamplifier ORTEC142 and an ORTEC570 shaping amplifier (shaping time: 0.5 µs). Pulse height processing, beam scanning and 2D map acquisition was carried out by a hardware and software system developed at the RBI [14]. We calibrated the electronic chain using a Si surface barrier detector and a precision pulse generator, in order to compare pulse heights provided by the reference Si detector with those from the diamond device. The spectral sensitivity of the IBIC set-up was ~1100 electrons/channel, corresponding to about 0.4% charge collection efficiency for 4 MeV He ions in diamond; the noise threshold level was set to channel 20 (7.2% CCE).

A similar experimental set up was adopted for IBIC measurements on sample 2. In this case, the ion beam scan area was about 300×300 µm$^2$.

### 3. Results

Fig. 3a shows the IBIC map collected from sample 1 by the horizontal (sensitive) electrode S$_0$, which is polarized at +80 V; the other vertical electrodes are grounded. Charge pulses overcoming the electronic threshold are generated only in the region surrounding the sensitive electrode; free carriers generated elsewhere do not induce detectable signals. The CCE profile along the horizontal direction centered in the middle of the horizontal electrode is nearly flat, with a small bump at the edge, followed by a rapid fall to zero (Fig. 3b).

When the applied bias is inverted (Fig. 4a), the IBIC map shows a complementary aspect, with no pulses detected in proximity of the sensitive horizontal electrode; detectable pulses are generated in proximity of the four vertical grounded electrodes, as highlighted by the CCE profile (Fig. 4c; solid curve) evaluated along the same direction as in Fig. 3b.

Fig. 4b shows the IBIC map obtained under the following bias configuration: $V_0$=-80 V and the other electrodes with a gradually increasing potential ($V_1$=-60, $V_2$=-40, $V_3$=-20, $V_4$=0 V, respectively). The relevant CCE profiles at the centre of the horizontal electrode are shown in
Fig. 3: (a) IBIC map from sample 1 collected under the following bias conditions: $V_0=+80$ V; $V_1=V_2=V_3=V_4=0$ V. The sensitive electrode is $S_0$ (ref. schematics in Fig. 2). The CCE is encoded in the gray scale shown on the bottom right, which represents the median of the CCE distribution in each pixel. (b) Horizontal CCE median profile along the rectangular region highlighted in the IBIC map.

Fig. 4c (dashed curve). In comparison with the case of all the vertical electrodes grounded (IBIC map in Fig. 4a), the profile shows a more pronounced decrease of the maxima as the distance from the sensitive electrode ($S_0$) increases.

The IBIC theory provides a satisfactory interpretation of these results [9]. If we assume a perfect intrinsic (i.e. no doped) material with ideal ohmic contacts, the traditional approach based on Ramo’s theorem, which consists in two different steps, can be adopted. First, the weighting potential is mapped by solving the Laplace equation assuming a unit potential at the sensitive electrode while all the other electrodes are grounded. This allows us to identify the region where charge pulses are generated, whose intensity is proportional to the difference in the weighting potentials between the initial and final position of the moving charges [15]. The second step consists in evaluating the carrier trajectories. This can be accomplished considering only the drift mechanism of transport (i.e. diffusion is neglected) and solving the Laplace equations with the actual bias potentials at the electrodes.
Fig. 4: (a) IBIC map from sample 1 collected under the following bias conditions: $V_0=-80$ V; $V_1=V_2=V_3=V_4=0$ V. (b) IBIC map collected under the following bias conditions: $V_0=-80$ V; $V_1=-60$ V; $V_2=-40$ V; $V_3=-20$ V; $V_4=0$ V. In both cases, the sensitive electrode is $S_0$ (ref. schematics in Fig. 2). The CCE is encoded in the gray scale shown on the bottom right, which represents the median of the CCE distribution in each pixel. (c) Horizontal CCE median profile along the rectangular regions is highlighted in the above IBIC map (a) (solid line) and (b) (dashed line), respectively.

Figs. 5a, 5b and 5c show the weighting potential maps relevant to the sensitive electrode $S_0$ evaluated by solving numerically Laplace’s equation by means of Finite Element Method (FEM) [1]. The streamlines indicate the trajectories of positive charges subjected to the actual electrostatic field generated by the bias configurations relevant to Figs 3a, 4a and 4b, respectively. The generation point was set at a depth of 8 µm below the surface, which corresponds to the maximum of the Bragg ionization curve depicted in Fig. 1.

A comparison of the experimental IBIC maps with the corresponding theoretical potential maps highlights the role of electrons and holes in the charge induction.
Fig. 5: Weighting potential maps and electric field streamlines relevant to the IBIC maps shown in Fig. 3a (a), Fig. 4a (b), Fig. 4b (c) and Fig. 6a (d), respectively. The weighting potential is encoded in the colour scale in the middle of the figure. The streamlines are generated at a depth of 8 µm, corresponding to the maximum of the ionization curve. Arrows indicate the electric field at the generation points.

In Fig. 3a the signals are generated in proximity of the anode; electrons provide a negligible contribution to the CCE since they are rapidly collected by $S_0$ and cross a region with a nearly constant weighting potential. On the other hand, holes drift towards the grounded electrodes and span regions with weighting potential ranging from almost 1 to 0 (see Fig. 5a). If the charge generation occurs near the grounded electrode(s), the dominant contribution should be provided by electrons; however the CCE profile in Fig. 3b is null, indicating that the drift length (and thus the lifetime) of electrons is significantly shorter than the distance between the electrodes.

This interpretation is further corroborated by the results shown in Fig. 4a, where the bias polarity has been inverted. In this case, holes generated at the anodes (vertical electrodes) move towards the sensitive electrodes. Their drift lengths are sufficient to span regions with noticeably different values of the weighting potential (Fig. 5b).

It is worth comparing the streamlines in Figs. 5b and 5c. Due to the decreasing applied bias potentials at the electrodes, some trajectories generated at $V_2$ converge at $V_1$, spanning almost constant weighting potential values. This is the cause of the difference of the CCE profiles shown in Fig. 4c and relevant to the bias conditions of Figs. 4a and 4b.
Fig. 6: (a) IBIC map and relevant horizontal profile of sample 1 with graphitic buried electrodes under the following bias conditions: \( V_0 = V_2 = V_4 = 0; V_1 = V_3 = -100 \) V. The sensitive detector is \( S_2 \) (see schematics in Fig. 2a). (b) IBIC map and relevant horizontal profile of sample 2 with interdigitated metallic electrodes on the surface; the sensitive comb is grounded and the other is at -100 V (see scheme in Fig. 2b).

Fig. 6a shows the IBIC map and the relevant horizontal profile collected by the second vertical electrode (\( S_2 \)) under the following biasing conditions: \( V_0, V_2, V_4 \) grounded and \( V_1, V_3 \) at -100 V. As expected, only the region in proximity of the sensitive electrode (anode) provides a detectable signal induced by hole transport towards the closer cathodes; the profile is Gaussian-like with a maximum of about 60% occurring at the centre of \( V_2 \) and a FWHM of about 30 \( \mu \)m. Fig. 5d shows the relevant weighting potential map and hole trajectories.

It is interesting to compare such a profile with the profile obtained in similar bias conditions on sample 2, which was equipped with interdigitated metallic electrodes deposited onto the diamond surface. Fig. 6b shows the relevant IBIC map, which highlights the comb-like structure of the sensitive electrode. The CCE profile shows that at the centre of each tooth-comb, the efficiency reaches a CCE of 80%, a value remarkably higher than that observed in Fig. 6a. The reason of this difference can be attributed to the fact that above-mentioned “cap layer” region has been subjected to a sub-threshold damage induced by ion irradiation. The obtained data indicate that after annealing the diamond structure has not been fully recovered [16, 17].

From the ionization curve in Fig.1, it is apparent that about 25% of the carriers are generated in the cap layer. Assuming that in this region the residual damage is still active in inducing efficient hole trapping, we can reasonably expect a reduction of 25% of CCE with respect to the pristine material as assumed to be the sample 1.

4. Conclusions
In this paper we report on the IBIC characterization of a CVD diamond ionization radiation detector with buried graphitic electrodes fabricated by DIBL.

From the point of view of the material qualification, IBIC maps acquired under different polarizing bias conditions have evidenced that induced charge signals are formed mainly at the anodes, whatever are the bias polarization conditions and/or location of the sensing electrode. The interpretation of such a fact is based on the assumption that the material is intrinsic and the electrical contacts are ohmic. Such an assumption is supported by the fact that the currents between the electrodes are below the detection limit, whatever are the bias polarities. The experimental results are hence compatible with a model which considers holes as the dominant carrier responsible for the induced charge formation, as already observed by other authors by the analysis of the evolution of the induced transient current [18]. The low contribution of electron transport can be then ascribed to trapping/detrappping effects [19, 20], resulting in a signal rise time higher than the integration time (0.5 µs) of the electronic chain.

In order to strengthen our interpretation of the results of IBIC experiments, we performed a FEM analysis based on the adjoint equation approach [21, 22]. From a numerical point of view, we defined the device characterization as the set of parameters declared in the simulation that enabled it to reproduce the experimental findings. Assuming standard values for carrier mobility [1], horizontal CCE profiles in Figs. 3, 4 and 6 were reproduced assuming a constant hole lifetime of 0.4 ns in the bulk material (comparable with that found in [1]), and a value for the electron lifetime of the order of 25 ps. In order to model sample 1, both electron and hole lifetimes were set equal to 25 ps in the cap layer (Fig. 1). The results reinforced our interpretation of the induced charge pulses based on the negligible electron contribution.

From the point of view of the fabrication process qualification, it has been shown that the layer above the graphitic channels shows traces of a residual damage induced during ion micromachining, which was only partially healed by thermal annealing. Residual trapping centres are therefore present, which strongly reduce the carrier (hole) lifetimes (few ps), making the cap layer (few micrometer beneath the surface) almost inactive for the detection of ionizing radiation.

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