Nuclear Instruments and Methods in Physics Research A 738 (2014) 119–125

Contents lists available at ScienceDirect

Nuclear Instruments and Methods in Physics Research A

journal homepage: www.elsevier.com/locate/nima

Beta particles sensitivity of an all-carbon detector

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ARTICLE INFO

Article history:
Received 17 June 2013
Received in revised form
18 November 2013
Accepted 8 December 2013
Available online 16 December 2013

Keywords:
Polycrystalline diamond
Graphite contacts
2D arrays
CCE
Beta particles detectors

ABSTRACT

The response of high quality polycrystalline diamond pixel detectors to ^{90}Sr beta particles is reported. Laser induced surface graphitization was used to realize 36 conductive contacts with 1 mm × 1 mm area each, pitch 1.2 mm, on one detector side whereas a 8 mm × 8 mm large area graphite contact was realized on the other face for grounding or biasing. A proximity board was used to hold the matrix, the amplifiers and to bond nine pixels to test homogeneity of response among 36 detector pixels. Two configurations were used to test charge collection uniformity and signal dependence on voltage. Both configurations showed noise pedestal fitted with a Gaussian curve of 1150 equivalent electrons (1σ) and typical beta source particles spectrum. Reversing the bias polarity the pulse height distribution does not change and the saturation of most probable value of charge collection was observed around ±200 V (0.4 V/μm) with reasonable pixel response uniformity equal to a most probable value 1.28 ± 0.05 fC. The charge collection efficiency (CCE) measurement was implemented using coincidence mode acquisition with an external trigger made by a commercial polycrystalline diamond slab. The detector shows a CCE = 0.59 estimated using the 1 mm^2 large graphite pixel. The information earned with this first prototype will be used to design the new board with amplifying electronics for reading all 36 pixels at a time and perform experiments with monochromatic high energy electrons.

1. Introduction

Diamond is considered one of the most promising materials to monitor high flux photon beams, synchrotron radiation and high energy physics charged particles beams like the ones produced at the LHC. Nowadays it is possible to produce very low roughness (2–4 nm in average) large area polycrystalline (~25 cm^2) and epitaxial (~50 mm^2) substrates for the fabrication of detectors [1,2] and high performance electronic devices [3,4]. Due to the high strength of the covalent bond (347 kJ mol^{-1}) a large amount of energy is required to remove a carbon atom from the diamond lattice, making it very hard also to displace an atom in the diamond structure. The energy needed to displace and atom in this very compact lattice has been evaluated with theoretical and experimental methods [5–7] and values higher 45 eV per atom were reported. The high cohesive energy is also responsible for the higher thermal conductivity (22 W cm^{-1} K^{-1}) in the crystalline diamond and small thermal expansion coefficient at room temperature (0.8×10^{-6} K^{-1}). Such mechanical characteristics [8] reflect on the electronic properties [9], qualifying the material as strong dielectric at room temperature (RT) or high temperature wide band-gap semiconductor. The resistivity in the dark is in excess of 10^{15} Ω cm, with large breakdown electric field (~2×10^7 V cm^{-1}) and high carrier mobility (2400 cm^2 V^{-1} s^{-1} for electrons, 2100 cm^2 V^{-1} s^{-1} for holes). Such values remain quite constant also for polycrystalline diamond [8] mainly depending on the grains dimension distribution and amount of defects at grain boundary. Therefore, as for other wide band gap semiconductors, the fabrication of good Ohmic contacts is not easy. Mechanical polishing, used to reduce the surface roughness, influences the characteristic of metals deposited to form electric contacts by pinning the Fermi level through the action of surface electronic states with the consequence to have injecting or blocking contacts at random. In the recent past, carbide forming metals have been the solution to obtain stable Ohmic contacts [10,11] whereas Schottky contacts have been obtained successfully both by surface hydrogenation [12] or boron deposition before metallization [13]. The interposition of a DLC layer by sputtering or implantation [14] has been also tested for injecting contacts.

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0168-9002/$ - see front matter © 2013 Elsevier B.V. All rights reserved.
http://dx.doi.org/10.1016/j.nima.2013.12.022

Besides the above-mentioned techniques to obtain stable Ohmic contacts, the ion-beam-induced graphitization of diamond has been extensively investigated [15–17]. The advantages of the latter approach consist in the possibility of obtaining conductive electrodes in an all-carbon environment [18–21], in the high thermal stability of the final structures, and in the possibility of defining contacts and electrodes into high-purity substrates without involving growth stages or complex lithographic processing. To this aim, also laser-induced graphitization has been extensively investigated [22–25] as a technique suitable for the diamond surface modification with the production of thin conductive layers. The laser treatment approach has been studied not only for graphitization but also for surface micromachining [26,27] and bulk diamond modification [28].

In a previous paper [29], we reported the preliminary study of the same matrix with graphitized pixels by using non-optimized readout electronics. Although reasonable pixels response uniformity was evidenced with good discrimination with respect to the noise pedestal, the rise and the peak of the beta spectrum was not visible. An evaluation about dependence of collected charge on the applied voltage, as well as the analysis of possible causes of poor observed spectra was not possible. Here we present the fabrication, the characterization and a discussion of results of the same 6×6 pixel array detector for nuclear particles monitoring fabricated onto a high quality polycrystalline slab with 1 mm × 1 mm area large sensitive pixels defined by UV laser graphitization.

The array response homogeneity, number of mean collected electrons, applied electric field dependence, noise and signal amplitude have been studied using two different biasing configurations. The charge collection efficiency of our polycrystalline slab has been evaluated implementing an external trigger configuration as usually done in literature, but using a second polycrystalline diamond on the back side of the board used to hold the graphitized matrix under test.

2. Technology issues

2.1. Materials

The diamond film has been grown on a silicon substrate in a mixture of methane, hydrogen and oxygen by using a plasma enhanced CVD deposition system operated at 5 kW microwave power and 2.45 GHz frequency. Typical growth conditions were as follows: 1.2–2.5% methane concentration in the mixture, total gas flow rate 1000 sccm, pressure 95–100 Torr, substrate temperature 820–850 °C. More details about deposition process can be found elsewhere [30]. The silicon substrate was etched away and a 10 mm × 10 mm slab cut from the diamond wafer using a frequency doubled Nd:YAG laser; then both nucleation and growth sides of the slab were mechanically polished and thinned to around 500 μm with average surface roughness of 2–8 nm. To remove graphite and metal residuals from the sample it was dipped in hot chromic acid solution ($K_2Cr_2O_7$, 1.6% weight, in H_2SO_4; 15 min at 120 °C) followed by aqua regia (25 vol% HNO_3 in mixture HNO_3+HCl; 10 min at 90 °C), rinsed in hot DI water (1 min at 80 °C), and then in isopropanol (1 min at 25 °C).

A polished electronic quality polycrystalline slab, 10 mm × 10 mm × 0.5 mm, from Element Six was used to fabricate a single pixel detector to implement the external trigger. Dipping into a hot organic solvent, with ultrasound assistance, was the first treatment aimed to remove polishing residuals from the substrate. Then both faces were oxygen terminated by O_2/Ar plasma (100 W, 50 mTorr) and 200 nm of silver sputter deposited through a shadow mask to define a 8×8 mm^2 area.

2.2. Graphitization

The surface was irradiated by a UV KrF excimer laser (CL 7100, Optosystems Ltd.) at a wavelength of 248 nm with pulse duration 20 ns. Before the irradiation the surface was covered with thin absorbing gold layer to prevent re-deposition of ablated carbon material on diamond surface outside of the irradiated area, which may induce short circuit between two adjacent graphitized pixels. When all graphitic pixels have been produced the sample was etched in aqua regia to remove the gold layer together with re-deposited carbon, so the space between pixels was clean. Few nanometers of graphite could be also removed from the pixel during this fast etching process. The laser was used in the projection optical setup, illuminating uniformly a square mask with a side of 4 mm. The reduced (by a factor of 20) image of the template was projected onto the sample surface by the objective. The size of illuminated region was 200 μm × 200 μm, the local laser fluence at the sample surface was 10 J/cm^2, well above graphitization threshold for polycrystalline diamond. Each 1 mm × 1 mm pixel on the large grain growth side was produced by laser spot scanning (two adjacent spots were overlapped for 20 μm, 3 laser pulses were sent in each spot), thus the pixel is composed of 36 spots 200 μm × 200 μm area each. The image of a single graphite pixel, where the superposition of the spots is apparent, is shown in Fig. 1. Color and intensity differences valuable in the picture are an artifact due to non-homogeneous illumination. These 36 pixels with a pitch of 1.2 mm occupied 7 mm × 7 mm area. Another 8 mm × 8 mm large graphite contact was shaped on the back nucleation side.

The laser irradiation produced a crater covered by graphitic layer, which thickness of 150–200 nm was evaluated on a reference surface from the drop of IR optical transmission or, alternately, by measuring with the optical profilometer (New View 5000, ZYGO) the crater deepening after graphite removal by oxidation in air at 600 °C. The relief on the crater's margin is shown in Fig. 2, revealing that the top diamond layer with the thickness about 500 nm has been ablated. Noticeable is the relief of superposition regions of neighboring 200 μm × 200 μm laser spots.

The Raman spectrum taken at 488 nm excitation wavelength (LabRAM HR 840, Horiba) for virgin polycrystalline diamond sample indicates only a single narrow diamond line (1332 cm^{-1}) as shown in Fig. 3. After laser irradiation there is no more diamond peak but D (1360 cm^{-1}) and G (1590 cm^{-1}) bands corresponding

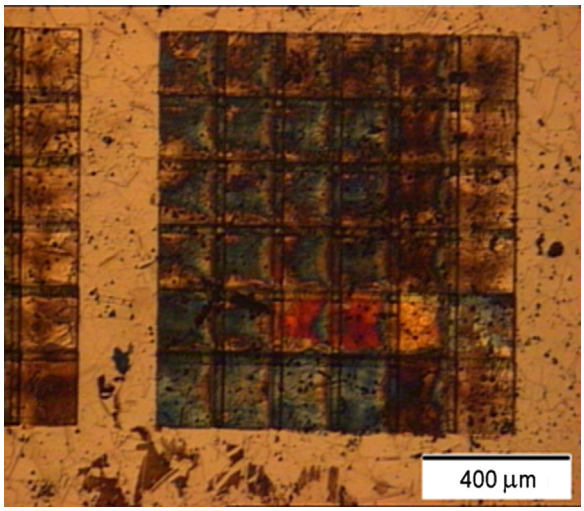

Fig. 1. Image of a single graphite pixel where it is visible the superposition of 36 spots.