Radiation Damage of Polycrystalline CVD Diamond with Graphite Electrical Contacts

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Abstract

In this work we show preliminary results of radiation damage for a polycrystalline diamond with graphite contacts in terms of time response after 62 MeV protons irradiation for a total fluence of $(2.0\pm0.08)\times10^{15}$ protons/cm². In addition, we describe the realization of a new type of device made with graphite micro-strips by laser micro-writing on diamond surface. In this way we made 20 graphite micro-strips of width about 87 µm and spacing between each other of about 60 µm.

Introduction

Due to its exceptional electrical, thermal and optical properties, diamond is a very attractive material for radiation detection. Diamond can detect any kind of radiation that is more energetic than its band-gap of 5.47 eV, e.g., deep UV photons, X-rays, gamma rays, charged particles and neutrons with a dynamic range in energies spanning from 5.47 eV up to GeV of cosmic rays. Because of its radiation hardness it needs no frequent replacements, it can be operated at room temperature with no need for cooling, it has a resistivity several orders of magnitude greater than silicon, an extremely low leakage current and no need for p- type or n-type junctions as required in the fabrication of silicon radiation detectors.

In this work, we show preliminary results of a device based on detector grade polycrystalline CVD diamond film with graphite electrical contacts fabricated on

both faces of the diamond surfaces (called "Sandwich" configuration). The device was prepared by front and back irradiation of the diamond bulk by a 193 nm UV ArF excimer laser according to the experimental setup in ref. [1]. This device was characterized as a nuclear radiation detector and consequently we evaluated its response under irradiation by a 60 Co γ -ray source and 120 GeV proton beam [1].



Fig. 1: Setup for micro-focusing laser beam. We used an aluminum mask with a square hole (1) and a beamsplitter (2) to divide the laser beam in two parts. One of these parts was directed onto a micro-spot focusing objective (3) to focalize the beam on diamond sample surface (4) that was kept on an automatic moving holder (5).

In this work we studied radiation damage of the diamond detector in terms of time response in order to verify radiation damage of the new type of ohmic electric contact. Finally, we made graphite micro-strips on another diamond sample surface to realize a new type of device by the experimental setup in Fig. 1.

Diamond graphitization

In this work we used ArF excimer laser (193 nm) for diamond surface graphitization because of the optical properties of high purity diamond that absorbs radiation at wavelength of about 193 nm and it is transparent at radiation having longer wavelength [2]. The absorbed laser energy is converted to lattice thermal energy making the transition diamond to graphite energetically favorite [3]. In the same time laser light power density and laser shoots are kept lower than diamond ablation thresholds [4], [5].

In literature, graphitization processes with ArF laser light on CVD diamond are reported [5]. The authors showed that a graphitic layer was created, and not another allotropic carbon structure, making use of micro-Raman scattering spectroscopy. In ref. [6] we also made use of this technique on thermal grade polycrystalline CVD diamond to confirm the graphitic nature of the fabricated layers by excimer laser. The micro-Raman measurements on the sample irradiated with ArF excimer laser showed the presence of trigonally coordinated carbon (graphite) with different degrees of structural disorder associated to the G band at about 1580 cm^{-1} , to the D (disorder) band around 1350 cm⁻¹ and to the G' band at about 2720 cm^{-1} , due to the so called turbostratic t-graphite. The turbostratic t-graphite is a stacking of graphene layers which are rotationally random with respect to one another along the c axis. The G' band of a typical turbostratic t-graphite is a single Lorentian peak, like in a graphene monolayer, but with

a FWHM value between 50 and 75 cm⁻¹. This is a larger value than the FWHM of a graphene monolayer G' band, which is about 25 cm⁻¹ [7]. From the results obtained in previous work we graphitized an high quality Chemical Vapor Deposition (CVD) diamond sample (Fig. 2) by an ArF excimer laser and we used the same experimental conditions of ref. [6].



Fig. 2: Optical microscopy image of the polycrystalline CVD diamond detector grade film of $(5 \times 5 \times 0.3) \text{ mm}^3$ size after laser treatment. The black pad at the center of the device is the photo-generated graphite electrical contact created on the diamond sample surfaces taken by ref. [1].

Micro-strip graphitization

We realized graphite micro-strips on high quality Chemical Vapor Deposition (CVD) diamond sample surface by micro-focusing ArF excimer laser beam (193 nm). The sample was an un-doped synthetic detector grade polycrystalline diamond acquired from Diamond Detectors Ldt. The thickness was of 300 µm after mechanical-chemical polishing and laser cuted to a size of 0.5×0.5 cm². In detail we prepared 20 graphite micro-strips by irradiation of the diamond surface with a 193 nm UV ArF excimer laser (Lambda Physik LPX305i). The laser emitted 20 nsec long light pulse with an energy of about 160 mJ/pulse at 10 Hz repetition rate. The laser beam with a transverse size of about 20×10 mm² was directed onto an aluminum mask with a square hole to select only the central part of the beam. The mask was the object and we focalized an image on diamond surface by a micro-spot focusing objective, 15X for 192 -194 nm wavelengths and a numerical aperture (NA) that was 0.32. Before the objective we used a fused silica coated beamsplitter designed for a low transmission ratio of approximately 10-30 % at 193 nm at 45 degrees to reduce the laser power. The beamsplitter divides the laser beam in two parts. One of these parts was directed onto the micro-spot focusing objective to focalize the beam on diamond sample surface that was kept on an automatic moving holder. The holder was a x-y handling stage automatically controlled by a 2D step motors (see Fig. 1). The focused image was a circle of about 87 um in diameter and the local laser fluence was about 5 J/cm^2 at the sample surface. The irradiation intensity was very uniformly distributed within the radiation spot. The device was treated in air and at room temperature. Moving diamond sample at a velocity of about 0.3 mm/s we produced 20 graphite micro-strips with width of about 87 μm and spacing between each other of about 60 μm (Fig. 3).



Fig. 3: Optical microscopy image of the polycrystalline CVD diamond detector grade film of $(5 \times 5 \times 0.3) \text{ mm}^3$ size after laser treatment. The black strips are graphite micro-strips on diamond sample surface.

On the other surface of diamond sample we made a scansion of the surface moving the diamond holder to a velocity of 0.3 mm/s and we created a graphite pad of 9 mm² like in Fig. 2 using the same experimental setup

used for micro strip focusing (see Fig. 1). The laser light power density and laser shoots are kept lower than diamond ablation thresholds [4], [5] in each case. By the sample in Fig. 3 we have demonstrated that it's possible to realize micro-strips on diamond sample. By theoretical considerations we expected this results. Graphitization process is activated from heat transfer on diamond surface by laser light absorption. Laser used by us emitted 20 nsec long light pulse, i.e. we transfer heat on diamond surface inside a time interval of 20 nsec. If we give to diamond surface heat from an epicenter (laser spot) the heat diffuses from it toward the external points. If we multiply the time duration laser pulse with thermal diffusivity for diamond given to us by Diamond Detectors Ltd company (2.8-11.6 cm²/s) we have the value of the graphitized surface by our own nanosecond laser (0.714-23.2 μm²). This value is much less than the surface graphitized by us, because we focalized a laser spot with a diameter of 86.67 µm to create the micro-strips.

Radiation damage

In this section we complete the study about diamond detector grade [1] by studying radiation damage of polycrystalline diamond in terms of time response. For this purpose we irradiated the detector by an estimated integrated fluence of $(2.0\pm0.08)\times10^{15}$ protons / cm² at an energy of 62 MeV on diamond sensor and evaluated the response under β source after and before protons irradiation. For protons irradiation we used the experimental setup of Fig. 4.



Fig. 4: Setup in the experimental area for the 62 MeV proton irradiation at INFN Laboratori Nazionali del Sud (Catania, Italy).

We collected protons from the zero degree beam line of the INFN Laboratori Nazionali del Sud Superconducting Cyclotron. The nominal beam current was of 10 nA and the protons bunch of about 3 nsec width and 25 ns period. While for β analysis we employed the setup in Fig. 5. A commercial diamond [8] were used as reference detector respect to graphitized detector. We lined up the devices and irradiated with the same β source to ensure a generic β electron across diamond under test (graphitized diamond) and go toward diamond to trigger (reference commercial diamond) as we can see in Fig. 5.



Fig. 5: β source experimental setup with fast charge amplifiers to study fast detector response.

We used two fast charge sensitive amplifiers (gain = 8 mV/fC, time rise = 2 ns, time pulse = 7 ns, noise = 450 e⁻/pF) and a digital scope to record the output signals from the devices. In Fig. 6 we report the β time responses before and after protons irradiation. The rise-time of the peaks is dominated by front-end electronics, but no change in pulse shape is observed before and after irradiation for both detectors and consequently no change in time response is evident.

Conclusions

In this work we continued the studies about a device based on detector grade polycrystalline CVD diamond film. In ref. [1] we obtained the electrode thicknesses (44 ÷ 83) nm by resistivity measurement on

graphite layers, we demonstrated the Ohmic behavior of the electrodes by dark current measurements made on the diamond device. Finally, we demonstrated that this device produce a response under irradiation by a ⁶⁰Co γ -ray source and 120 GeV proton beam. In this work we showed preliminary studies of radiation damage for polycrystalline diamond in terms of time response and realized that before and after 62 MeV protons irradiation for a total fluence of (2.0±0.08)×10¹⁵ protons/cm² diamond doesn't change in time response if we test the device with a β source. The last work was the fabrication of a new type of device made with graphite micro-strips. We didn't characterize yet this detector, but we demonstrated that it's possible to do microwriting on diamond surfaces by laser techniques. In this way we made 20 graphite micro-strips on diamond surface with width of about 87 µm and spacing between each other of about 60 µm.



Fig. 6: Time response to a β source for a commercial diamond detector before (a) and after (b) protons irradiation and for the graphitized diamond detector before (c) and after protons (d). The insets report a zoom of the peaks.

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