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Registration of high-intensity electron and X-ray fields with polycrystalline CVD diamond detectors

Aleksey E. Bolotnikov

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DISCLAIMER

Registration of high-intensity electron and X-ray fields with polycrystalline CVD diamond detectors

L.N. Davydov*[a], A.V Rybka[a], A.A. Vierovkin[a], S.F. Dudnik[a], V.I. Gritsyna[a], V.E. Kutny[a], O.A. Opalev[a], V.A. Shevchenko[a], I.N. Shlyahov[a], V.E. Strelnitsky[a], A.Eh. Tenishev[a], V.L. Uvarov[a], R.B. James[b], A.E. Bolotnikov[b], P.M. Fochuk[c]

[a]National Science Center KIPT, Kharkov, Ukraine 61108;

[b]Brookhaven National Laboratory, P.O. Box 5000 Upton, NY 11973-5000;

[c]Chernivtsi National University, Chernivtsi, Ukraine 58012

ABSTRACT

We developed radiation-hard diamond detectors for registering intense fields of high energy electrons and X-rays, and monitoring the mode of operation of electron accelerators. After synthesizing a diamond film of detection quality up to 350-microns thick by chemical vapor deposition (CVD), we analyzed it by infra-red spectroscopy. We also developed techniques for heat treatment of the film, chemical etching, substrate removal, contact application, and priming by an exposure to X-rays and electrons. This work supported the production of detectors with a specific resistance of 10^{14} Ohm×cm. The dependence of the detector signal's amplitude on the displacement voltage was investigated under exposure to a direct electron beam with a current ranging from 660 to 930 mA. The duration of the leading edge of a detector pulse was 5 μs. Experiments also were undertaken on the registration by diamond detectors of Bremsstrahlung radiation with an end-point energy of 9 to 70 MeV. We also evaluated the dependence of the amplitude of the detector's signal on the displacement voltage. Our comparison of detectors' physical properties and detectors' response to alpha-particle irradiation before and after the exposure to the accelerator beam showed no degradation, even after the absorbed dose exceeded 11.5 MGy.

Keywords: CVD-diamond, detector, electron radiation, X-rays, accelerator

1. INTRODUCTION

Together with impressive progress made in the instrumentation of high-energy and accelerator physics, the search constantly continues for more effective, radiation harder and more temperature resistant materials for radiation detectors. The most intensive work focuses on semiconductor solid-state devices that are in demand for both fundamental studies and various radiation technologies. For example, in promising photonuclear technologies (e.g., for the soft production of isotopes, processing of radioactive waste, and activation analysis) fields of high-energy ($E \geq 10$ MeV) and high intensity ($> 10^3$ W/cm^2) photons are needed.

Radiation- and nuclear-technologies now are being developed at NSC KIPT, using, in particular, intense sources of Bremsstrahlung photons obtained via the conversion of a high-current electron beam at linear accelerators. Detector technology is needed for measuring the space distribution of the radiation intensity in a plane normal to the beam line, as well as along it. Taking into account the high intensity of the radiation, the realization of direct measurements was problematic. As discussed in this paper, this ability was obtained after a wide-gap semiconductor (an artificial CVD polycrystalline diamond film) was synthesized at NSC KIPT.

Among the wide-gap materials suitable for radiation sensors a special place belongs to diamond as a semiconductor material with the widest energy gap (5.4 eV) known. It possesses the further attributes of high-radiation hardness, nearly tissue equivalence ($Z = 6$ against $Z = 7.1$ for human tissue), and high carrier mobility, along with the ability to function at room temperature and higher. The rapid progress made in manufacturing detector-grade artificial diamonds in the last two decades, for example [1-3], both in a poly- and single-crystal form, compensated for its main drawback (i.e., the material's high cost).

*ldavydov@kipt.kharkov.ua phone 38 057 335-6513; fax 38 057 335-2683; www.kipt.kharkov.ua

In Table 1 several characteristics of conventional semiconductors used for radiation sensors (Si and GaAs) are compared with those for selected wide-gap semiconductors (CdTe, SiC, and C-diamond).

Table 1. Energy gap (E_g), static dielectric susceptibility (ε_0), Debye temperature T_D, melting temperature T_m, electron (μ_n) and hole (μ_p) mobilities, displacement energy E_d and minimum energy E_0^{min} of electron necessary for creation of a displacement defect for some semiconductors [4-6].

Semiconductor	E_g, eV (0 K)	ε_0	T_D, K	T_m, K	μ_n, $cm^2/V \cdot s$	μ_p, $cm^2/V \cdot c$	E_d, eV	E_0^{min}, keV
Si	1.17	11.7	645	1690	3000	500	11-22	115-330
GaAs	1.52	12.9	344	1510	8500	420	9.0; 9.4	230; 260
CdTe	1.6	10.3	200	1371	1200	80	5.6; 7.8	235; 340
SiC	2.86	10.2	1200	3073	230	70	25	
C (diamond)	5.4	5.7	2240	4300	1800	1400	80	530

The goal of our work was to create and investigate a CVD diamond detector for high-energy electron beams and photon radiations, and develop methods for monitoring the operation of electron accelerators, as well as other dosimetry applications.

The creation of detectors made from artificial diamond films faces several serious problems connected to the processes for growing diamonds, and the development of detector devices. The most serious difficulty in the CVD synthesis of diamond films was the necessity to lower the level of inclusions and structural defects that hinder our obtaining material of needed electrical quality. It also was essential to develop a technique for, and to conduct tests on the detector's radiation hardness under intensive electron- and Bremsstrahlung-fluxes at electron accelerators LU-10 and LU-40 in NSC KIPT [7,8].

2. SPECIMENS' PREPARATION AND EXPERIMENTAL TECHNIQUE

Polycrystalline diamond films (PDF) for fabricating detectors were synthesized by a method of chemical deposition from a vapor phase (CVD). An original method of working-gas activation with a glow discharge stabilized by magnetic field was developed [9]. This type of discharge ensures the injection of a high specific power in the discharge volume (up to 150 W/cm^3), that creates PDFs of large size, up to 1 cm^2 and thicknesses of 300–350 microns with lower power inputs. Fig. 1 presents a typical structure of the film's growth side consisting of the pyramidal grains with square and rectangular cross-sections. The transverse size of the crystallites is up to 100 microns.

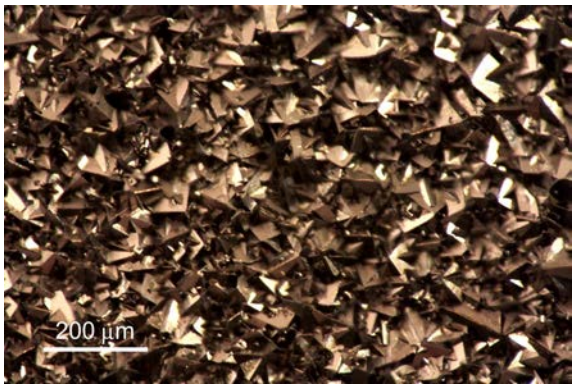

Figure 1. Polycrystalline diamond film grown on a silicon substrate.

We investigated the quality of the as-grown diamond film by infra-red spectroscopy. Fig. 2 shows the IR absorption coefficient of PDF samples obtained in our process of consistently improving the growth procedure. The samples were synthesized in a carbon-hydrogen mixture and then detached from a silicon substrate. For comparison, the absorption coefficient of natural diamond with a low level of impurities is also indicated in this figure [10]. Our progress in bettering the quality of the growing CVD diamond film allowed us to synthesize diamond films with good detector quality and use them not only for sensors of X- and gamma-rays, but also for charged particles (electrons, alphas, protons), and neutrons.

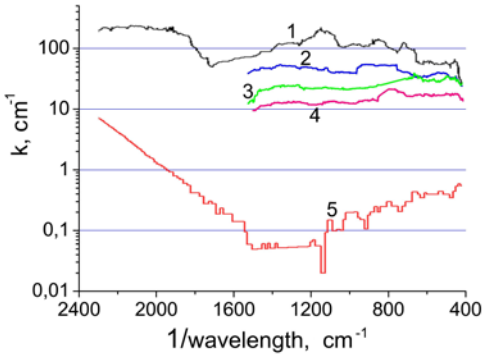

Figure 2. IR absorption coefficient of polycrystalline diamond films. Samples 1, 2, 3, and 4 were synthesized from a carbon-hydrogen mixture, 5 is natural diamond with a low level of impurities [10].

For manufacturing the ionizing-radiation detectors, we developed procedures for heat treatment of PDF films, chemical etching them, removing them from substrates, and priming by an exposure to radiation (X-ray and electrons); we formulated practices for applying metal contacts, bonding the wires, and packaging the detectors.

The study of films' physical properties started with our measurement of the current-voltage characteristics. As a displacement voltage source and a measuring device, we employed a pico-amperemeter Keithley 6487. Fig. 3 depicts the modifications of the current-voltage characteristics of PDF film processed sequentially by annealing in the air, chemical etching, and the exposure to electrons. It is evident that each treatment differently modifies the film's electro-physical properties. The most noticeable increase in the resistivity occurred after annealing in air at 600ºC. The effect of priming of the detector material by exposing it to 2.5-MeV electrons was evident after an absorbed doze of 1.5 kGy. Concurrently, the resistance of the diamond film increased further. This processing resulted in achieving a specific resistance of "self-carrying" (detached from the substrate) PDF samples equal to 10^{14} Ohm×cm.

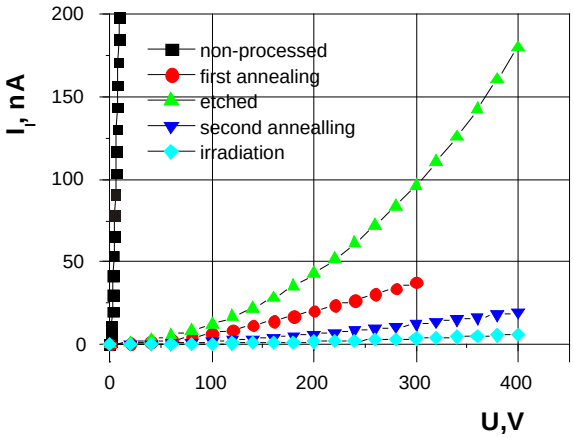

Figure 3. Current-voltage characteristics of CVD diamond after different stages of treatment.

For investigating the radiation performances of PDF films we created electrical contacts of various geometry on their surface. Planar metallic contacts (Ti, Al, Cr) in form of in-between-connected strips were applied on the film's growth surface and substrate surface by a method of ion-plasma sputtering in electric-arc vacuum installation. However, the best charge collection efficiency of non-equilibrium carriers was obtained with detectors having coplanar interdigitated contacts. Fig. 4 illustrates such Al contacts made on growth side of the PDF film by conventional photolithography. The contact layer was 0.8-microns thick, while the strip's pitch and width were 0.3 mm.

After we applied the contacts, we determined the efficiency of the detectors for the registration of α particles. This was accomplished by measuring the pulse-height distribution of the detector signal while irradiating it with α particles from an uncollimated ^{239}Pu source. The spectrometric measuring system consisted of a preamplifier, a shaping amplifier

Canberra 2025, high-voltage source Canberra 3106D, and a 12-digit ADC. The displacement voltage varied between 100–500 V.

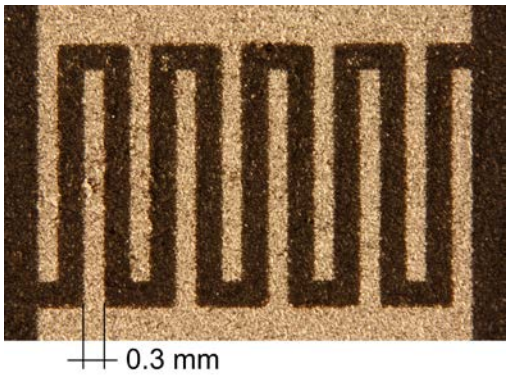

Figure 4. Interdigitated Al contacts on the growth surface of the diamond film.

The pulse-height distributions of the detector signal under α irradiation from ^{239}Pu source are shown in Fig. 5. Using this data, the displacement voltage (U_d) dependence of the total number of the registered pulses was obtained (Fig. 6); these results testify to the dosimetry prospects of our CVD diamond detectors.

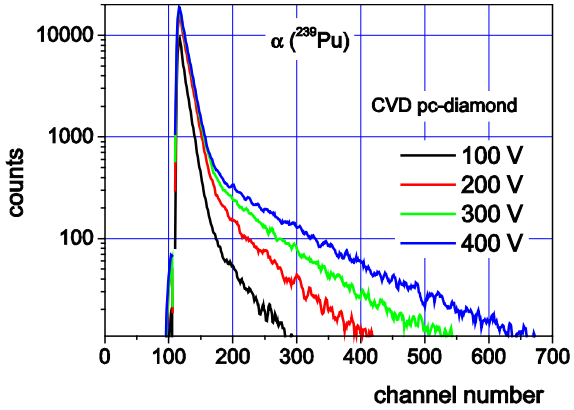

Figure 5. Pulse-height distributions of the detector signal from ^{239}Pu α-source for different displacement voltages.

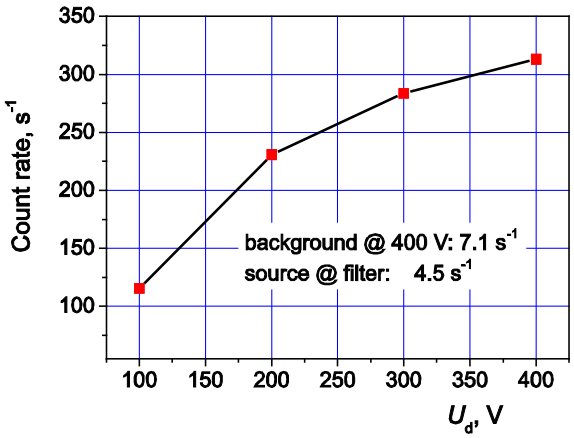

Figure 6. Displacement-voltage dependence of the pulse count-rate of the diamond detector exposed to α particles.

3. STUDY OF RADIATION HARDNESS

Radiation hardness usually is understood as an invariance of the parameters of the material when it is exposed to different kinds of radiation. The larger the absorbed dose needed to degrade the detector parameters, the harder the semiconductor material is to radiation.

For potentially radiation-hard materials, we refer to semiconductors with large binding energy, such as diamond, boron nitride, silicon carbide. For characterizing this bonding, we apply a parameter of the threshold displacement energy E_d (see Table 1), viz., the minimum energy that should be transmitted by a particle incident to the semiconductor's lattice for forming a Frenkel pair (vacancy and interstitial atom). Semiconductor materials with strong bonding and high E_d and having a wide gap (Table 1) present an alternative to silicon for monitoring applications in harsh radiation environments. Indeed, the exposure of silicon dosimeters to 6-MeV electrons to doses of 3 Gy at dose rate of 2-7 Gy/min decreases their sensitivity by 35 % [11].

We investigated the radiation hardness of the CVD diamond detectors at the electron accelerators LU-10 and LU-40 (NSC KIPT, Ukraine). First experiments were performed at LU-10 under a direct (not scanned) electron beam with the following parameters: Electron energy 9 MeV, pulse current 800 mA, and accelerator pulse rate 4 Hz. Figure 7 is the scheme of the accelerator output device. It consists of the graphite collimator C with a 15-mm diameter orifice placed axially symmetric to the beam line. The investigated detector, D, is positioned close to this orifice, and a graphite Faraday cup FC was placed behind it. The collimator and Faraday cup are loaded with resistors of 51 Ohm. The detector's load resistance is 100 kOhm. The detector displacement voltage varied between 10–100 V with an increment of 10 V. As a displacement voltage source, we used the power supply unit B5-49. The amplitude and duration of pulses at the collimator, Faraday cup, and detector were measured with the digital oscilloscope, TDS1012.

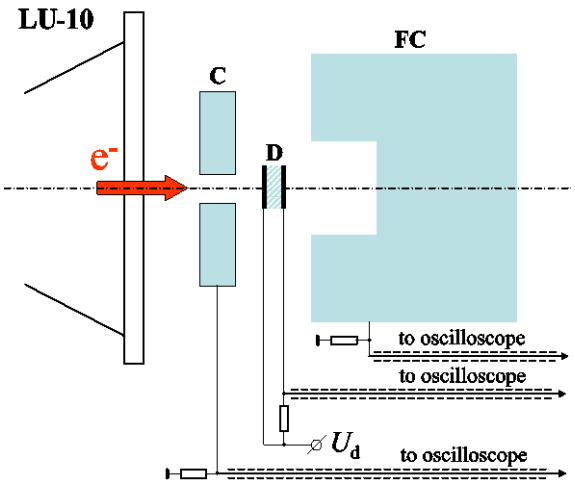

Figure 7. Scheme of electron-beam registration at the accelerator LU-10.

The oscillogram of the detector signal at the moment of accelerator pulse transmission is shown in Fig. 8. The oscillograms of a signal from the Faraday cup together with the diamond detector's signal (at displacement voltage 80 V), are shown in Fig. 9.

From Fig. 9, it is evident that the duration of the leading edge of the detector signal is 5 μs and of the trailing edge 3 ms in response to an electron beam with pulse-width of approximately. 3 μs. The trailing edge is so protracted due to the delay in the long transmission line. Figure 10 illustrates the displacement voltage's dependence of the amplitude of the detector signal excited by the electron beam. The Faraday cup's signal had the amplitude of –30 V corresponding to a pulse current of 588 mA and the collimator signal's amplitude of –15 V corresponding to a current of 294 mA.

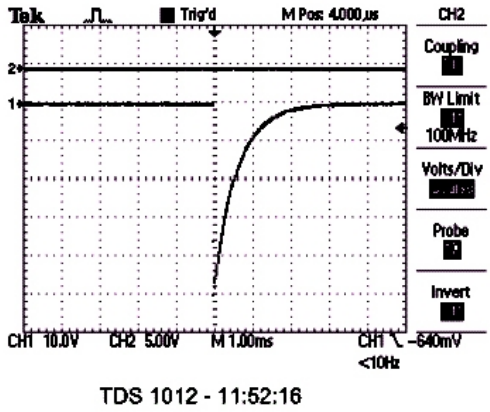

Figure 8. The diamond detector's signal at a displacement voltage of 80 V.

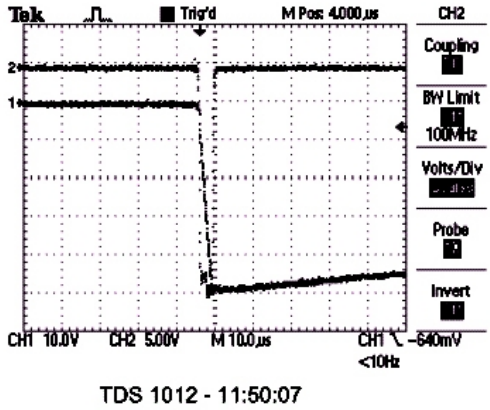

Figure 9. Signals from the diamond detector (channel 1) and Faraday cup (channel 2); U_d = 80 V.

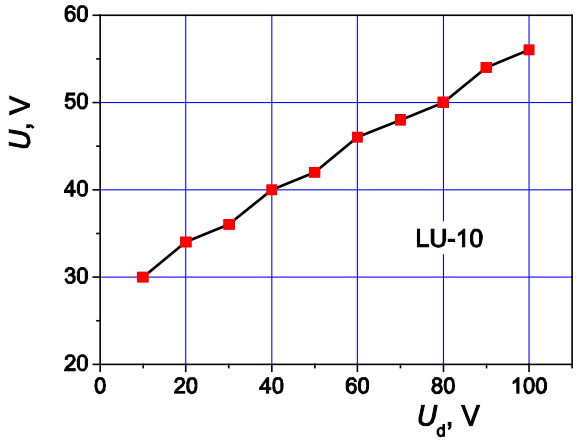

Figure 10. Dependence of the detector signal amplitude on the displacement voltage (pulse current of 800 mA).

The accelerator LU-10 ordinary was engaged in a technological program with a special conveyer with containers holding irradiated products and Red Perpex dosimeters, slowly moving across the scanned beam in front of the detector. The oscillogram in Fig. 11 illustrates the diamond detector's signal when the last container leaves the irradiation zone.

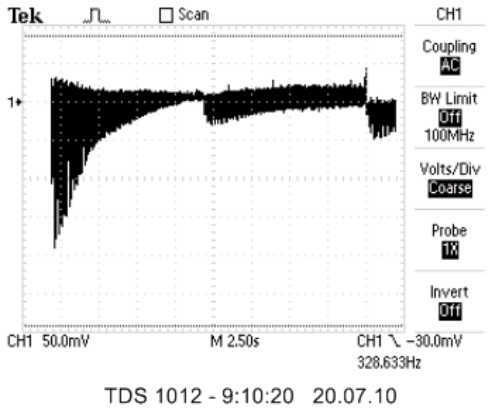

Figure 11. Oscillogram of a diamond detector's signal when the last container leaves the irradiation zone; displacement voltage is 50 V.

To investigate the response of the detector to electron beams of different intensity, we carried out experiments at two different pulse-heights of the accelerator beam current: 880 mA and 660 mA and the same pulse-rate, 4 Hz. Figure 12 shows the dependence of the amplitude of the detector's signal on the displacement voltage and the accelerator pulse current, while exposed to the direct electron beam at the LU-10.

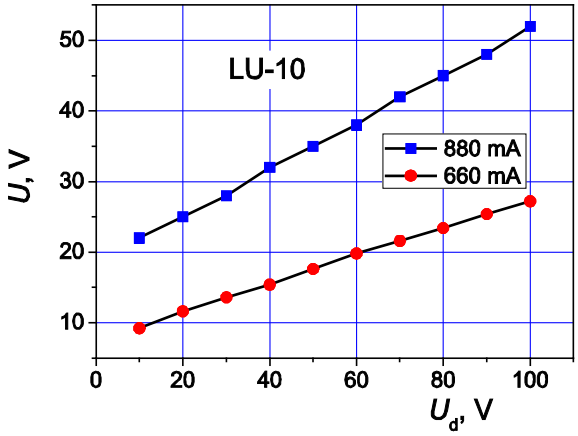

Figure 12. Displacement-voltage dependence of the voltage pulse of the detector signal for two values of the pulsed accelerator current.

For determining the detector's service life under electron irradiation, we raised the accelerator LU-10 pulse rate to 250 Hz with a sweeping electron beam. The electron energy was 9 MeV, and the average current was 770 μA. The average dose rate at the detector's position was 2 kGy/min. The exposure was done in two intakes with a 4-day break because of technical reasons. The integral exposure time was about 96 hours, and total absorbed dose amounted to 11.5 MGy.

The condition of the detector was estimated by the amplitude of the output pulse. The displacement voltage was 50 V, and the load resistance was 100 kOhm. Despite its intensive exposure and the high dose received by the detector, the amplitude of its signal hardly changed. Accordingly, we verified that the detectors made of polycrystalline diamond have high radiation-resistance, and that, together with dosimetry tasks, they can be also used for monitoring the accelerator-beam's energy.

4. REGISTRATION OF BREMSSTRAHLUNG AT ACCELERATORS LU-10 AND LU-40

Further we investigated the detector's response while exposed to high-energy X-rays. Our first experiments with the mixed electron- and X-ray radiation were conducted at the accelerator LU-10. To obtain photons, a 5-mm thick steel converter was positioned in front of the collimator. The Faraday cap (FC) registered the residual electron flux. The

amplitude of the Faraday cap's signal was 1 V corresponding to a current of 19.6 mA; its amplitude did not changed when the current of the electron beam was changed. The measured dependences of the voltage pulse of the detector signal on the displacement voltage and pulsed electron current during an exposure to X-rays at the accelerator LU-10 are illustrated in Fig. 13. The electron energy was 9 MeV; the pulse current 880 and 660 mA; and the pulse rate was 4 Hz.

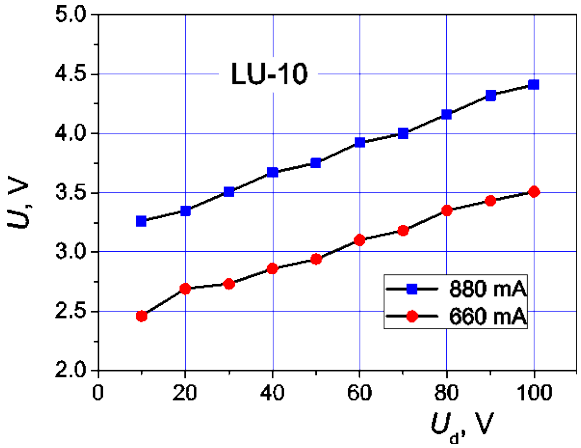

Figure 13. Dependence of the voltage pulse of the detector signal on the displacement voltage when exposed to Bremsstrahlung for two values of the pulsed accelerator beam current.

The next experiment was performed with the aim of investigating the performance of the diamond detector under the intensive flux of Bremsstrahlung free of the electron component. Accordingly, we positioned a Ta converter cooled by running water in the trajectory of the electron beam. The converter was irradiated by the scanned beam along its surface. The layout of this experiment is shown in Fig. 14. Parameters of the LU-10 electron beam were the following: Electron energy 8.8 MeV, pulse current 930 mA, pulse rate 250 Hz, scanning rate 3 Hz, and scanning width 35 cm.

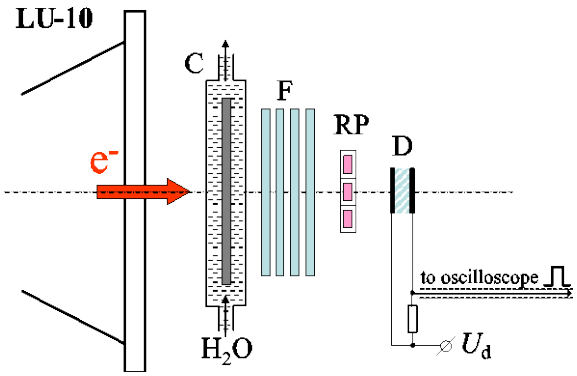

Figure 14. Scheme of the Bremsstrahlung registration at the accelerator LU-10.

Behind the converter C, a filter F consisting of four 2-mm Al sheets was positioned to suppress the residual electron current. Following the filter, at distance 20 mm, we placed the Red Perpex 4034 dosimeters, RP. The diamond detector, D, was positioned closely behind the dosimeters RP. The displacement voltage, U_d, was applied to the detector, and the detector's output was connected to the digital oscilloscope.

The measurements were taken at the moment of incidence of the photons on the detector. Their spectral maximum lies at about 0.1 of the end-point energy (8.8 MeV). The dosimeters RP after 8 minutes have shown a dose 40.7 kGy, so the dose rate was 85 Gy/second. Figure 15 shows the dependence of the voltage pulse of the detector's signal on the displacement voltage; the corresponding oscillogram of the detector's signal at 100-V displacement voltage is shown in Fig. 16.

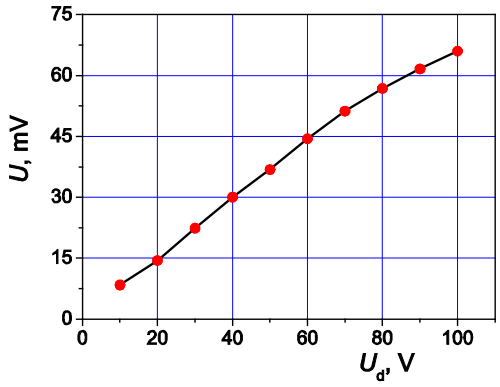

Figure 15. Dependence of the detector pulse signal on the displacement voltage while under X-ray irradiation at accelerator LU-10.

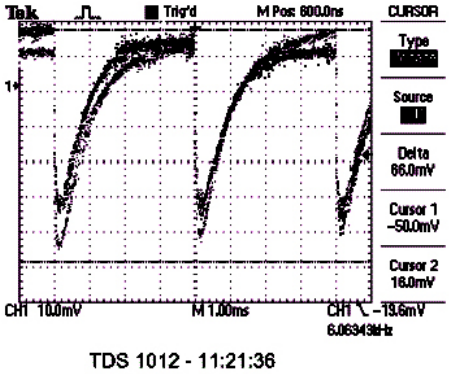

Figure 16. Oscillogram of the detector signal during an exposure to X-rays (displacement voltage 100 V).

After three runs, the total dose absorbed by the diamond detector had attained 200 kGy. Before and after an exposure to X-rays, we investigated the current-voltage diagrams (Fig. 17) and the response of the detector to alpha-particles (Fig. 18). Curves 1 and 2 in Fig. 17 correspond to the leakage current through the detector's volume, curves 3 and 4 to the current between interdigitated surface contacts. These diagrams testify that the detector's contacts had and retained their Ohmic character after 200 kGy of X-ray radiation.

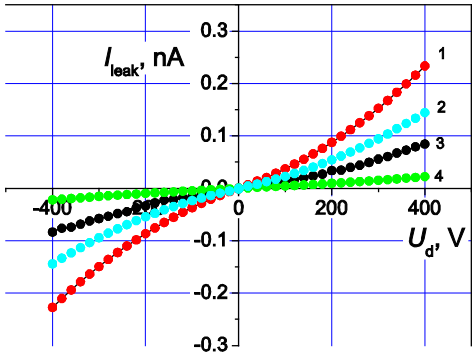

Figure 17. Current-voltage dependences of the detector before (1, 3) and after an exposure (2, 4) to an X-ray dose of 200 kGy.

The comparative investigation of the physical properties and response of the detector to alpha-particles from a ^{239}Pu source conducted before and after an exposure at the accelerator also revealed the absence of degradation of the detector after the absorbed dose exceeded 200 kGy (Fig. 18). A small amelioration of the charge collection observed after an irradiation is due to the priming effect of the detector's material.

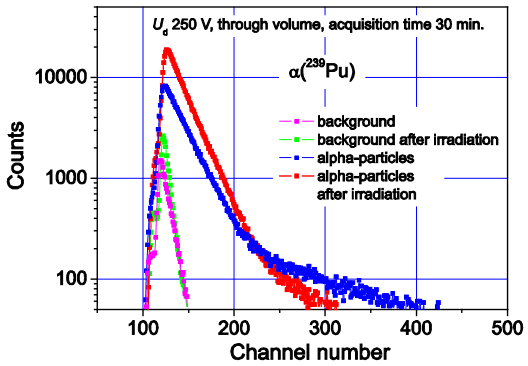

Figure 18. Pulse height distributions of the detector signals before and after the exposure to an X-rays dose of 200 kGy at the accelerator.

To explore the operation of diamond detectors at higher X-ray energies, we undertook experiments at the accelerator LU-40. The layout of the experimental equipment is shown in Fig. 19. The accelerator operated in two regimes with the following parameters of the electron beam: (1) Electron energy 52.5 MeV, pulse current 65 mA, beam pulse rate 50 Hz; and, (2) electron energy 70 MeV, pulse current 53.5 mA, and beam pulse rate 50 Hz.

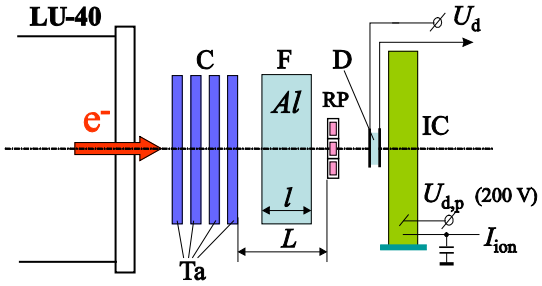

Figure 19. Scheme of the Bremsstrahlung registration at the accelerator LU-40.

Just against the outlet window of the accelerator, we placed a composite converter C containing four Ta plates each 1-mm thick. The converter was cooled with air. Behind the converter we positioned an Al cylindrical filter F for suppressing the residual electron current. The filter's diameter was 50 mm, whilst its chosen length depended on the energy of the electrons: l = 70 mm for 52.5 MeV, and l = 90 mm for 70 MeV. Next to the filter, at a distance L=100 mm from converter, the diamond detector D was situated with the RP dosimeters in front of it, and ionization chamber IC at some distance behind it.

The dose rate was 20 kGy/h for the 52.5-MeV beam and 25.7 kGy/h for 70 MeV. The detector's load resistance was 100 kOhm. The detector displacement voltage varied within 10–100 V, with an increment of 10 V (Figs. 20 and 21).

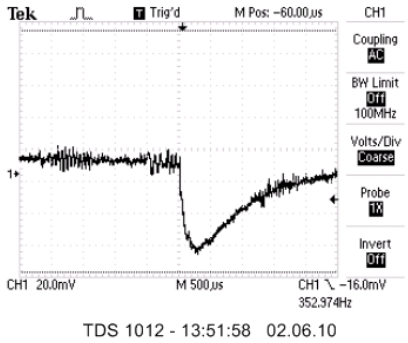

Figure 20. Signal oscillogram of the diamond detector irradiated by Bremsstrahlung X-rays. Displacement voltage 100 V. Electron beam energy 52.5 MeV.

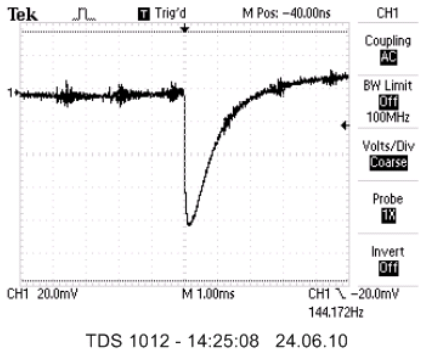

Figure 21. Signal oscillogram of the diamond detector irradiated by Bremsstrahlung X-rays. Displacement voltage 100 V. Electron beam energy 70 MeV.

Figure 22 presents the dependences of the amplitude of the detector signal on the displacement voltage obtained in this experiment for two values of electron energy (52.5 MeV and 70 MeV).

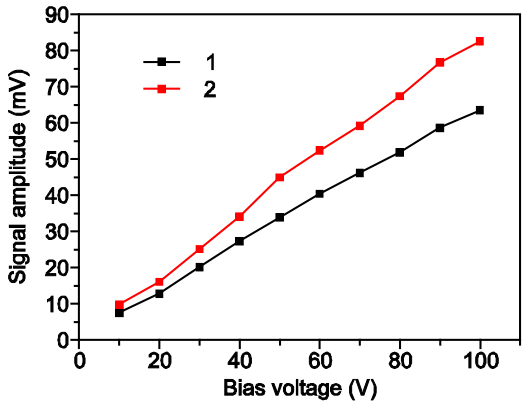

Figure 22. Dependence of the pulse amplitude of the detector signal on the displacement voltage exposed to Bremsstrahlung X-rays for two values of electron beam energy (1) 52.5 MeV and (2) 70 MeV.

Undoubtedly, detectors made of polycrystalline diamond have high radiation resistance and can be used for dosimetry and monitoring of the accelerator beam's energy.

5. CONCLUSIONS

Our studies show that the radiation hardness of our manufactured CVD polycrystalline diamond detector is high. It is connected to a nature of the detector material – polycrystalline diamond. Irradiation by electrons and high-energy photons, unlike neutron irradiation, does not induce collision cascades and creates predominately point defects (vacancies and interstitials) that, however, may evolve into more complex formations but only at higher temperatures. Ionization processes, i.e. excitation of electronic shells and formation of pairs of charge carriers, are the main contribution to energy losses. Radiation defects are unstable at the temperatures of the irradiation at the accelerator; therefore, the processes of defects relaxation take place under their thermal annealing. This explains the outstanding radiation hardness of the manufactured CVD diamond detectors. Summarizing, we conclude the following.

1. A detection-quality diamond film, 350-microns thick was synthesized by chemical deposition from a vapor phase (CVD). The investigation and our choice of regimes of thermal annealing, chemical etching, irradiation and metal contacts deposition allowed the production of detectors with a specific resistance of 10^{14} Ohm×cm.

2. The displacement voltage dependences of the detector signal amplitude were investigated during an exposure to a direct and a swept electron beam of energy of ~ 9 MeV and different beam currents, as well as during an exposure to Bremsstrahlung X-rays at different energies of the electron beam (9, 52.5, and 70 MeV). The X-ray dose rates were correspondingly, 5 kGy/min, 20 and 25.7 kGy/h. All these dependences showed almost linear behavior in all studied

range of displacement voltage up to 100 V. The width of the leading front of detector pulse makes 5 μs, in response to a 3 μs-wide electron beam pulse.

3. Our testing demonstrated that the detectors have high radiation hardness and can be successfully used for dosimetry and monitoring of intensive fluxes of electrons and X-ray radiation. The comparative studies of the detectors' I-V characteristics and their response to radiation taken before and after irradiation testified to the absence of observable degradation after an absorbed dose of 11.5 MGy.

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