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# PARTICLE DETECTOR WITH DIAMOND SENSITIVE ELEMENTS GROWN IN A CUBIC HIGH PRESSURE APPARATUS

**Introduction.** Diamond is one of the most suitable materials for particle detectors, due to a wide band gap and a high radiation resistance of this material. However, diamond sensitive elements for detectors remain rare and expensive, which hinders their widespread use in nuclear physics and medicine.

**Problem Statement.** In recent years, new technologies for growing HPHT diamonds in cubic presses have been developed. They have allowed obtaining up to 50 high-perfection single crystal diamonds weighing up to 10 carats in one cycle. However, the physical properties of HPHT diamonds grown in modern presses have not been sufficiently studied, and the results of the application of such diamonds in ionizing radiation detectors have been unknown so far.

**Purpose.** The development of a particle detector with sensitive elements based on HPHT diamonds grown in a cubic high-pressure apparatus and the study of its characteristics.

*Materials and Methods.* Growing diamonds by the temperature gradient method in a cubic high-pressure apparatus. Particle detector. Irradiation with alpha particles.

**Results.** The temperature gradient method has been used to grow 7-9 mm diamond single crystals in a cubic high-pressure apparatus, and 0.4 mm thick diamond plates have been made of cubic and octahedral growth sectors. The physical properties of the samples have been studied. The detector amplifier has been developed, and the detector has been tested when irradiated with alpha particles. The results have showed reliable detection of ionizing events caused by alpha particles and the registration of induced pulses with an amplitude of 70-200 mV.

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**Conclusions.** The particle detector with diamond plates made of cubic growth sectors of HPHT diamonds grown in a high-pressure cubic apparatus from the Fe-Ni-C growth system when irradiated with alpha particles has showed the ratio of full width at half maximum of pulse (FWHM) of about 1 ns, which corresponds to the world's best analogs of diamond detectors.

Keywords: particle detectors, HPHT diamond, and nuclear electronics.

For a long time, diamond has been considered one of the most suitable materials for the sensitive elements of particle detectors. First of all, this is due to a wide band gap and a high radiation resistance of this material. An overview of various stages of the study of diamond detectors has been presented in [1-5]. At the early stages of research on diamond detectors, natural diamonds were used as sensitive elements. However, the natural diamonds suitable for detectors are rare and therefore highly expensive. With the development of methods for producing synthetic diamonds, more attention is paid to detectors based on diamonds grown by chemical vapor deposition (CVD) or temperature gradient at high pressure and high temperature (HPHT) methods. The characteristics of diamond detectors critically depend on the type of diamond defects and the content of impurities in the crystal [6–10]. Currently, attention is mainly focused on the CVD diamonds in detectors because of their availability on the market and relatively high quality. However, many researchers have pointed out that the HPHT diamonds have better physical and technological properties [11-14].

However, until recently, methods for growing diamonds in conventional devices such as *Leopard* or *Toroid* allowed obtaining only 3–5 diamond crystals per cycle of synthesis, which led to their high cost and limited use in detectors. In recent years, methods for growing diamond single crystals in cubic presses have been developed. This enables growing up to 50 high-quality diamonds in one cycle of synthesis [15–17]. Such technologies look promising to obtain affordable diamonds for detectors. However, the physical properties of HPHT diamonds grown in modern cubic presses have not been sufficiently studied, and the results of the use of such diamonds in detectors have been unknown so far.

## Synthesis of Diamonds and Production of Plates

To grow diamond single crystal samples, we use a CS-VII type cubic press with a plunger diameter of 560 mm. Figure 1 illustrates the main components of a standard cubic press that consists of six anvils connected to independent hydraulic cylinders pressing on cubic growth cell. This configuration makes it possible to evenly apply pressure to all surfaces of the cubic container, enabling the creation a pressure of up to 6 GPa in the growth cells of a much larger volume as compared with other types of presses. Typically, the CS-VII type presses are used for the production of diamond powders by spontaneous synthesis. For growing large diamond single crystals in the CS-VII presses by the temperature gradient method, a special container with a growth cell has been designed. This provides uniform pressure distribution and allows distributing temperature in such a way as to create a temperature gradient of a given value for growing 5-10 carat crystals at a rate of 5-7 mg/h [17].

Iron-based solvents, including Ni and/or Co, are commonly used to grow HPHT diamonds [18].



*Fig.* **1.** Cubic high-pressure apparatus: a – general scheme; b – section by planes of vertical symmetry. **1** – base plate (steel); 2 – backing plate (steel); 3 – cooling channel; 4 – cooling and fastening ring (steel); 5 – punch (hard alloy); 5 – growth cell



*Fig. 2.* Diamonds of the Fe-Ni-C growth system grown in cubic HPA: a - single crystals after partial etching; b - 0.5 mm thick plates cut from the crystal center in the plane (001)



*Fig. 3.* IR spectra of a diamond single crystal plate grown in the Fe-Ni-C system. Spectrum a - corresponds to the growth sector (111), spectrum b - corresponds to the growth sector (001)

To obtain a colorless diamond, alloying elements such as Al, Ti, Zr or Hf are added to the solvent alloy [19]. Our previous studies have shown that the IIa-type diamond single crystals cannot be used to make detectors of ionizing radiation from high-amperage currents. Therefore, for growing diamonds for detectors, Fe-Ni solvent alloy is chosen. The container is made of extruded pyrophyllite in the form of a cube with a side length of 58 mm. We use embryonic crystals with a size of 0.5 4 0.5 mm obtained by methods of spontaneous crystallization at a high pressure and temperature.

The typical size of the grown crystals is 5-7 mm (Fig. 2, *a*). From the central part of the three single crystals having clear-profile faces, 0.4 mm thick plates with a surface orientation (001) are cut as shown in Fig. 2, *b*.

The obtained plates have a clear visual identification of the growth sectors, which is typical for these crystals. The cubic growth sectors are almost colorless, while the octahedral growth sectors are colored yellow (Fig. 2, b).

To determine the content of nitrogen impurity, the IR absorption spectra in the cubic and octahedral sectors of diamond plate growth have been studied. A typical IR spectrum is given in Fig. 3. For all three samples the spectra are similar. The calculations have shown that for the spectra presented in Fig. 3, the concentration of nitrogen impurity in the form of paramagnetic C-centers is 4 ppm in the (001) growth sector and 50 ppm in the (111) growth sector.

For further studies, rectangular plates with a side length of 1.7-1.9 mm are cut from cubic and octahedral growth sectors. Both sides of the samples are covered with three layers of Ti / Pt / Au. The Ti layer is used to obtain a metal contact on the diamond; the Au layer is used to prevent oxidation of Ti and to connect with the contact wire. The Pt layer prevents diffusion between Au and Ti. After metallization, the diamond is annealed at 1073 K in Ar gas to create TiC on the diamond surface that provides an ohmic contact.

### **Current-Voltage Curves**

To assess the quality of contact coatings, the current-voltage curves (CVC) of the obtained samples of diamond plates with metallization have been analyzed. Figure 4 shows the dark I - V curves of the cubic (001) and the octahedral (111) growth sectors of plate No. 1. For the other two plates, the CVCs are similar. As one can see from Fig. 4, dark CVCs are fairly linear, which indicates a good contact resistance of diamond and titanium due to the formation of titanium carbide at the Ti-C interface.

According to Fig. 4, the dependences of the sample resistivity on the bias voltage have been calculated:

$$\rho = VS / Il, \tag{1}$$

where S is the contact area, l is the distance between the contacts.



*Fig. 4.* Dependence of dark current *I* on the electric field strength *E* applied to the contacts of the plane-parallel plate of the detector made of cubic (001) and octahedral (111) sector of HPHT diamond growth



*Fig. 5.* The resistivity  $\rho$  of the cubic (001) and octahedral (111) sectors of HPHT diamond growth in the dark mode as a function of the electric field strength *E* 

The results of the calculations are shown in Fig. 5. The dependence of the resistivity on the bias voltage is significantly nonlinear in the region of small values of V. This is due to a slight nonlinearity of the dependence I (V), which is invisible in Fig. 4, but obvious in Fig.5, when small values of I are in the denominator of formula (1). Some nonlinearity of the CVCs in the low-voltage region is apparently caused by the formation of a small Schottky barrier at the Ti – C interface [13].

### **Detector with Amplifier**

The scheme of the detector head and its design are presented in Fig. 6. The body of the head is made of aluminum alloy; the printed circuit board is made of foil microwave material, type FAF-4D. The diamond plates with applied electrodes are



*Fig. 6.* The head of a diamond elementary particles detector: a – electrical circuit; b – general view



Fig. 7. General view of the detector head and preamplifier

placed on gold-plated pedestals with through holes for alpha radiation passing through the board and the head body. The elastic current collectors hold the diamond plates and ensure their connection with the circuit elements. The head is equipped with two SMA Femeil connectors for supplying high bias voltage. The scheme and general view of the head of the diamond detector are presented in Fig. 6.

The detector amplifier uses Gali 52+ chips from Mini-Circuits based on InGaP heterogeneous bipolar transistors. The amplifier is made according to the manufacturer's recommendations. At a supply voltage of + 12V, the corresponding load resistor with a resistance of 150 Ohms and a TCCH-80 inductance choke, each stage has the following parameters:

- frequency range: from direct current to 2 GHz;
- signal amplification: 20 dB;
- noise factor: 2.7 dB at a frequency of 1 GHz; and
- useful current: 50 mA,

Three stages, separated by capacitors with a capacity of 1000 pF, provide a total gain of 60 dB



*Fig. 8.* Scheme of the experiment on recording the charge induced by alpha particles in a diamond detector

and a lower bandwidth of 3 MHz. Structurally, the amplifier is made similarly to the detector head. The general view of the detector head and the amplifier is shown in Fig. 7. The *Tektronics* MD03 oscilloscope is used to record the detector's response to alpha radiation.

### Irradiation with Alpha-Particles

A pressurized closed source of alpha radiation AZK244.28 manufactured by *Isotope*, in the form of a 5 mm high, 10 mm diameter cylinder (with a working window diameter of 6 mm) with a  $1.4 \times 108 \text{ s}^{-1}$  flow of alpha particles with an energy of the order of 5.2 MeV has been used as a source of ionizing radiation. The experiments are conducted in air environment, according to the scheme presented in Fig. 8.

The signal starts to appear when the electric field strength on the detector is ~  $1 \text{ V/}\mu\text{m}$ . The maximum applied voltage after which the breakdown begins is ~ 850-1100 V (E =  $1.5-2 \text{ V/}\mu\text{m}$ ).

The results of electrical pulse measurements on the right contact of the detector covers are shown in Fig. 9. The amplitude of the signal obtained from 5.5 MeV alpha particle and amplified by 40 dB at a ~1.2 V/µm electric field strength on the detector cover is ~ 80 mV, the full width at half maximum (FWHM) of the signal is about ~ 1 ns.

# Analysis of the Results

Since the analyzed diamond crystals grown in the Fe-Ni-C growth system differ in the minimum content of boron acceptor impurity and belong to *n*-type semiconductors, with a predominance of

nitrogen C-centers (see Fig. 3), the current of induced charges through the right contact of the detector (Fig. 8) associated with the mobility of electrons  $\mu_n(E)$  and the density of the induced charge *n* is defined by the ratio:

$$I(t) = qnl^2\mu_{\rm p}E,\tag{2}$$

where  $l^2$  is the contact surface area of the detector sensitive element.

This value is also determined by voltage pulse  $U_n$  caused by the charge induced by alpha particle, through relationship [20]:

$$I(t) = \frac{1}{R_{\rm in}A} \Big[ R_{\rm in} C_d \frac{dU(t)}{dt} + U(t) \Big].$$
(3)

It takes into account the gain A, the input resistance  $R_{\rm in}$ , and the total capacitance  $C_d$  (the sum of the detector own capacitance and parasitic capacitances relative to the detector body and its connection to the preamplifier) of the measuring system. Given that in our case  $C_{\rm d} < 5$  pF and the pulse duration  $\tau > 1$  ns, the first component in (3) may be neglected, and taking into account (2) we may write:

$$\frac{I_{\rm C}}{I_{\rm O}} = \frac{n_{\rm C}}{n_{\rm O}} \approx \frac{U_{\rm C}}{U_{\rm O}},\tag{4}$$

where the indices *C* and *O* denote the cubic and octahedral sectors of the diamond plate growth in the detector, respectively.

As can be seen from Fig. 9,  $U_{\rm C}/U_{\rm O} = 80 \text{ mV}/$ 60 mV  $\approx 1.3$  ( $U_{\rm c}$  and  $U_{\rm o}$  are signal amplitudes on detectors of cubic and octahedral crystals, respectively). Given (4), this means that the density of the induced charge in diamond samples cut from the cubic sector of crystal growth is by 23% higher that the charge density induced by diamond samples from octahedral growth sectors. In addition, the FWHM of pulse in the cubic sectors is 4 times less than that of the pulse recorded in the octahedral sectors. Since the mobility of charge carriers in diamonds significantly depends on the concentration of nitrogen [22], these differences in the parameters of detectors from different crystal growth sectors may be explained by different concentrations of donor impurities in the cubic



*Fig. 9.* Voltage pulse *U* recorded while a detector with diamond plate from cubic (*a*) and octahedral (*b*) sectors of diamond single crystal growth is irradiated

and the octahedral crystal growth sectors, as a result of which the mobility of majority carriers (the electrons) in the *n*-type diamond samples with a lower concentration of nitrogen (the cubic ones) is higher than in the samples with a higher concentration of nitrogen (the octahedral ones).

The obtained results have shown that the pulse FWHM in the case of the detector based on the sensitive element from the cubic growth sector of the HPHT diamonds synthesized in the Fe-Ni-C growth system irradiated with alpha particle is about 1 ns, which corresponds to the world best analogs of the CVD diamond detectors [21]. The concentration of nitrogen in the studied samples of the HPHT diamonds is higher as compared with the CVD diamonds. The obtained result is explained by more perfect crystal structure of the HPHT diamond.

In general, the technology for growing large HPHT diamonds in cubic presses followed by the manufacture of diamond plates from the cubic growth sectors look promising for obtaining sensitive elements of particle detectors. Further studies of detectors with such sensitive elements in medicine and high-energy physics are required.

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#### ДЕТЕКТОР ЕЛЕМЕНТАРНИХ ЧАСТИНОК З АЛМАЗНИМИ ЧУТЛИВИМИ ЕЛЕМЕНТАМИ, ВИРОЩЕНИМИ В КУБІЧНОМУ АПАРАТІ ВИСОКОГО ТИСКУ

Вступ. Алмаз є одним з найбільш придатних матеріалів для виготовлення детекторів елементарних часток, що пов'язано з широкою забороненою зоною та високою радіаційною стійкістю цього матеріалу. Однак алмазні чутливі елементи для детекторів залишаються рідкісними та вартісними, що стримує їхнє широке використання в ядерній фізиці та медицині.

**Проблематика**. Останніми роками розроблено нові технології вирощування НРНТ (*high-pressure high-temperature*) алмазів в кубічних пресах, що дозволяє отримувати до 50 монокристалів алмазу вагою до 10 карат з високою структурною досконалістю за один цикл вирощування. Однак фізичні властивості НРНТ алмазів, вирощених в сучасних пресах, досліджені недостатньо, а результати їх застосування в детекторах іонізуючих випромінювань невідомі.

**Мета.** Розробка детектора елементарних частинок з чутливими елементами на основі НРНТ алмазів, вирощених в кубічному апараті високого тиску, та дослідження його характеристик.

**Матеріали й методи.** Вирощування алмазів методом температурного градієнту в кубічному апараті високого тиску моделі CS-VII; детектор елементарних частинок; опромінення альфа-частинками.

**Результати.** Методом градієнту температур в кубічному апараті високого тиску вирощено монокристали алмазу розміром 7—9 мм, з кубічних та октаедричних секторів росту виготовлено алмазні пластини товщиною 0,4 мм. Досліджено фізичні властивості отриманих зразків. Розроблено підсилювач детектора та проведено його випробування разом з детектором при опроміненні альфа-частинками. Показано впевнене детектування іонізуючих подій, викликаних альфа-частками, з реєстрацією наведених імпульсів з амплітудою 70—200 мВ.

**Висновки.** Детектор елементарних частинок з алмазними пластинами, виготовленими з кубічних секторів росту НРНТ алмазів, вирощених в кубічному апараті високого тиску з ростової системи Fe-Ni-C, при опроміненні альфачастинками показав значення повної ширини на половині висоти імпульсу на рівні 1 нс, що відповідає кращим світовим аналогам алмазних детекторів.

Ключові слова: детектори елементарних частинок, НРНТ алмаз, ядерна електроніка.