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Development of particle detectors on the base of Minsk synthetic monocrystalline diamond

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Current situation with diamond detectors for particle physics applications is briefly described. Most important issues defining detector quality are enlisted. Improvement of detector quality due to thermobaric processing of initial diamond is described. Three samples of synthetic monocrystalline diamond detectors are described, one of them demonstrating unusual avalanche-like behavior.

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1. Introduction

Forward calorimetry at the International Linear Collider (ILC) requires extremely radiation hard materials. Absorbed dose here is expected to be up to 10 MGy per year. Diamond is the only material to date which was tested at such doses and remained operational [1], [2].

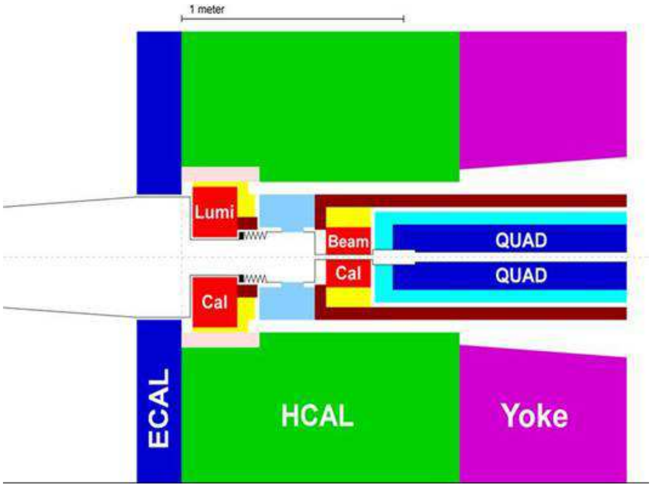

FIG. 1. Forward region of international linear collider. Annual absorbed dose here is expected to be up to 10MGy.

The main material tested currently for ILC forward calorimatry is polycrystalline CVD diamond. But there are serious obstacles hindering its application. Majority of samples tested in

DESY (mainly of Fraunhofer Institute production) develop excess currents with dose accumulation, up to microampere range in some cases (that is, one thousand times exceeding expected ionization current). Even the best samples (produced by Element 6, the spinoff enterprise of De Beers), free of this problem, demonstrate poorly predictable behavior in case of large dose rates and significant fluctuations of radiation environment. Response amplitude changes several times depending on the dose absorbed and the current dose rate [2]. It's hard to develop a calorimeter on the base of sensors whose response unpredictably changes at similar energy depositions. In the same time the only CVD-mono sample (also of De Beers production) tested in DESY behaves much better. Excess currents are absent, charge collection distance equals to the whole crystal thickness [3] thus not changing with the dose. As a result response amplitude unambiguously reflects energy deposition. So detectors on the base of synthetic monocrystalline diamond are still of great interest for the forward calorimetry of International Linear Collider despite their small dimensions. Monocrystalline diamond of Minsk production is considerably cheaper than CVD-mono of De Beers. Current article describes a particle detector based on Minsk monocrystalline diamond.

2. General principles and approaches in diamond detector development

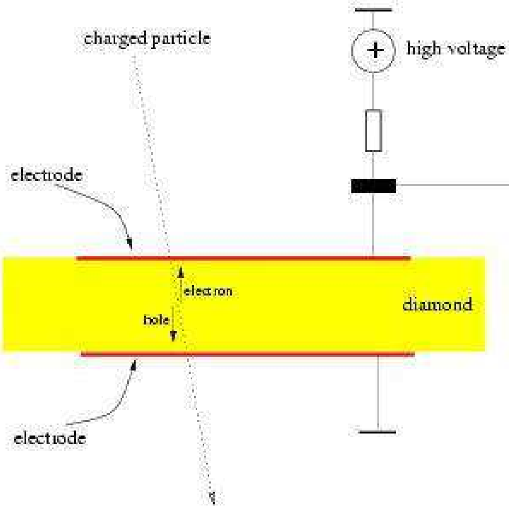

FIG. 2: Principle of action of diamond detector.

Wide bandgap of the diamond guarantees small leakage current (a fraction of picoampere) at usual detector dimensions without special techniques typical for silicon technologies (reverse biased p-n-junctions, guard rings etc.). An ideal diamond detector functions like a solid state ionization chamber, constituting a parallel-sided plate with metal electodes to create electric field. The real situation is more complicated because impurity atoms, inevitably present in diamond, are limiting the lifetime of free charge carriers, defective surface layer (produced during diamond slicing into plates) causes creation of undesirable space charge (which modifies the applied electric field), and metal-semiconductor contact constitutes Shottki diod in general case, which also leads to irregular field distribution inside the diamond plate. So the next conditions (at least) should be satisfied for creation of effective diamond detector: a) minimization of defects and impurities which reduce life time of free charge carriers; b) thorough surface processing to garantee removal of defective layer and absence of microcracks; c) special metallization procedure which provides creation of nonrectifying ohmic contact. Main impurities of the diamond synthesized with temperature gradient method are nitrogen and the metal used as a melted catalizer. Concentration of these impurities depends on the choice of the certain system of metals-catalizers, because both solubility of the metal in crystal and solubility of the nitrogen in metal varies widely for different metals. Furthermore, "gettering" is possible, that is binding of the nitrogen with special additives to reduce its final concentration in the crystal. Melted iron-nickel or iron-cobalt environments are mainly used at "Adamas" plant for the catalytic diamond synthesis. Nickel and cobalt have different solubilities in diamond. Moreover, they form impurity sites of the Me - X type, which have carbide or nitride nature, and differently influence the life time of free charge carriers. So the optimal choice of catalytic environment is one of the most important factors defining the final quality parameter of diamond detector - charge collection distance. Surface processing of the diamond plate before metallization requires special techniques. Standard silicon processing techniques (chemical-mechanical polishing in etching water solutions, for example) are impossible here due to extraordinary hardness of the diamond. One of rather simple methods of the chemical polishing of the diamond plates is etching in the melted nitre at 700°C. This method allows to etch several tens of nanometers, having thus removed rough defects. Thermochemical processing is a more sophisticated technology which consists of contact dilution of surface diamond layer in transition metals (Fe, Ni, Co etc.) at 700...1200°C and subsequent gasification of the carbon in hydrogen atmosphere. This method allows to reduce surface roughness down to Rz = 0.025μm. Deposition of carbide-forming metal sublayer is obligatory at metallization stage because it allows to resolve two problems at once: to provide good adhesion (because carbon for the carbide formation is taken out of the diamond bulk) and ohmic contact. Very important also is to prevent partial graphitization (carbidization is performed at high temperature) of crystal edges, otherwise large leakage currents can occur.

3. Monocrystalline particle detectors on the base of Minsk synthetic diamond

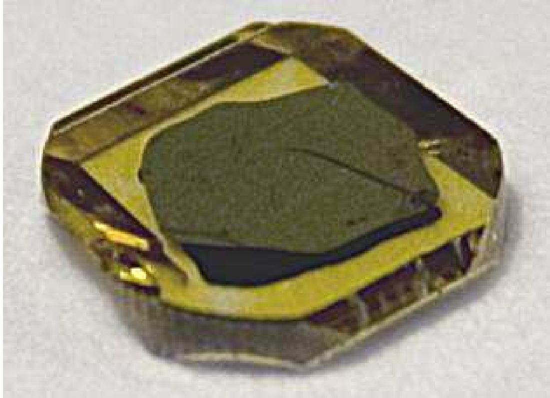

FIG. 3: Sample 171s1 tested as the first specimen of particle detector.

Parallel-sided plates of (100) (square) and (111) (triangle) orientation, cut out of synthetic diamond crystals, were used as initial samples. Initial crystals with mass 0.5 - 1.5 carat were grown at "Adamas" plant with temperature gradient method in Ni-Fe-C, Fe-Co-C (yellow) and Fe-Co-Al-C (white) environments in high pressure apparatuses of "split sphere" type [4]. Impurity composition of synthetic diamond crystal was investigated with optical absorbtion methods in the wavelength range 0.2 - 25 μm. Total impurity content (nitrogen, metals composing catalitic environment, boron) was measured to be $6 \times 10^{18} cm^{-3}$. Metallization was performed by thermal sputtering of titanium and gold with subsequent annealing for carbidization.

Three samples, 171s1, 219 and 77 were tested as particle detectors.

3.1. Parameters of the first sample

Currents were measured with picoampermeter B7-49. 15 minutes delay was given after each voltage step for the sake of transient current stabilisation, then current measurement was performed. Currents were measured without detector packaging, with help of contact device, depicted at fig.4.

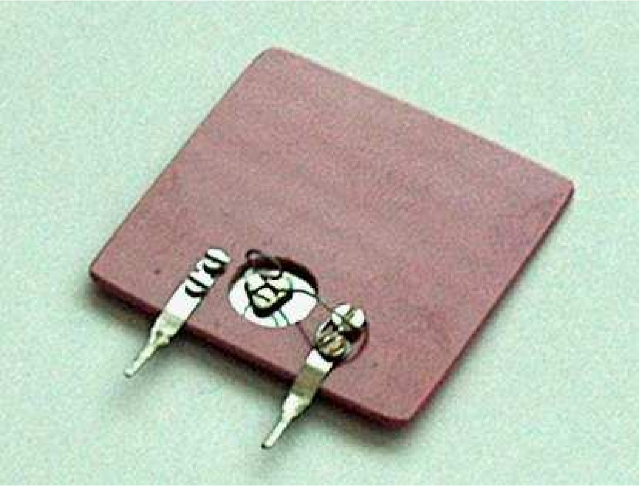

FIG. 4: Contact device for measurement of parameters of non-packaged diamond detectors.

I-V characteristic of our detector is shown on the fig.5. Subpicoampere currents are typical for diamond and prove absence of parasitic conductive films. Positive and negative branches are rather smooth and almost symmetrical, which confirms good quality of metallization.

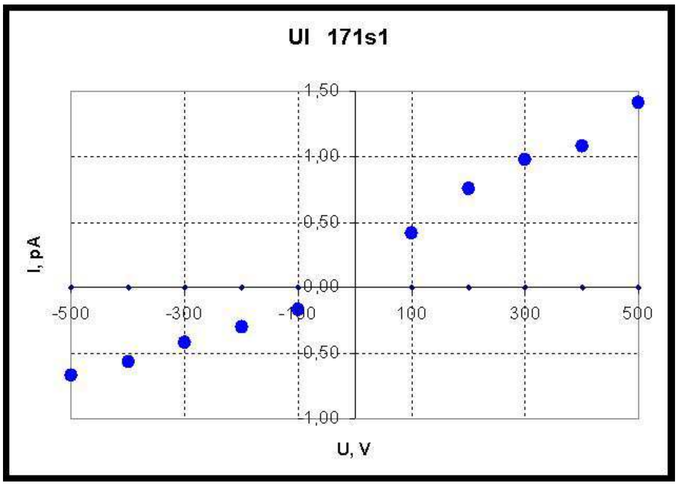

FIG. 5: I-V characteristic of the sample 171s1.

Oscillograms were taken with the help of DS03102A digital oscilloscope. The signal was read out with universal low-noise charge-sensitive amplifier "Tetrode", developed at NC PHEP BSU and produced at "Integral" plant, Minsk.

3.2. Detector on the base of thermobarically processed diamond

The main defects limiting life time of free charge carriers are impurity atoms. The main impurity in diamond is nitrogen. Majority of synthetic diamonds have nitrogen in replacing position (C-defect) thus belonging to type 1b. The most commonly used method of modification of diamond monocrystal defect structure is thermobaric processing, performed at conditions

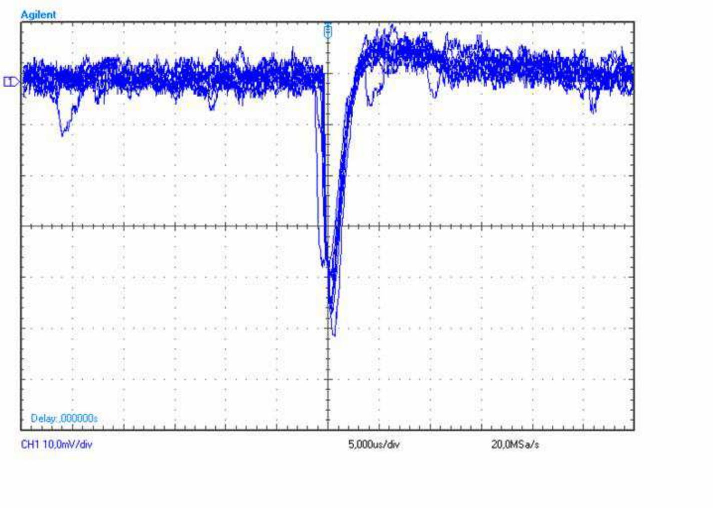

FIG. 6: Response to β-particles of the sample 171s1.

FIG. 7: Sample 219, thermobarically processed.

providing diamond stability. It's shown in [5], [6], [7] that conversion of C-form of nitrogen impurity into A-form is going on during annealing of 1b diamond at P = 6.0 - 6.5 GPa and T = 1700 - 2100°C.

So we have performed thermobaric processing of our samples using the same high pressure apparatus, which was used for the synthesis. Stabilizing pressure was 6.7GPa, temperature 1800°C, duration 4 hours.

Nitrogen defects of diamond lattice can be studied with absorption spectrometry in IR range. C-defect gives perfectly distinguishable signal at $1135 cm^{-1}$. A-defect gives a signal at $1282 cm^{-1}$. So it's quite possible to check the degree of annealing.

It is seen at fig. 8(a,c) that C-defects have been partially transformed into A-defect due to thermobaric processing. The degree of conversion is about 30 - 40%.

Five annealed samples behaved differently, the most pronounced positive effect was demonstrated by the sample 219. Its response to β-particles before and after annealing is shown at fig. 8(b,d).

3.3. Avalanche-like behavior of one of the samples

One of thermobarically processed samples (fig.9) demonstrated unexpected behavior. Response amplitude was 10 times larger then that of the samples 171s1 and 219. It was even larger then response of our reference sample (of De Beers production) which have charge collection distance comparable with the thickness of diamond plate. Response wasn't stable, and its practical significance is questionable. But one of possible explanations is an avalanche process

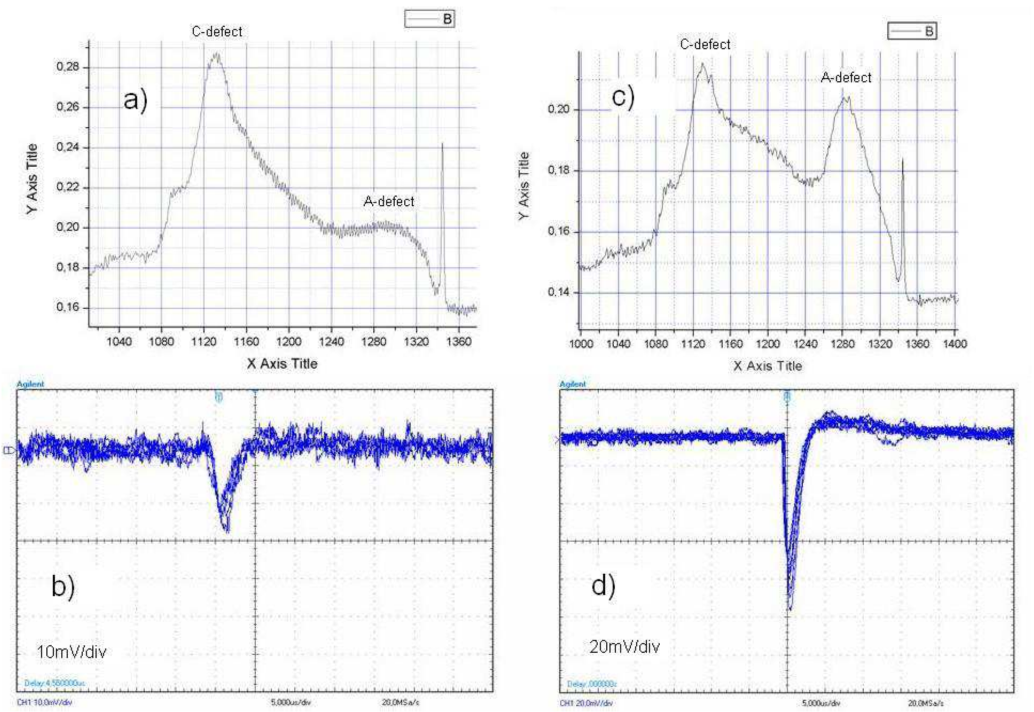

FIG. 8. IR spectrum and response to β-particles of the diamond before (a,b) and after (c,d) thermobaric processing.

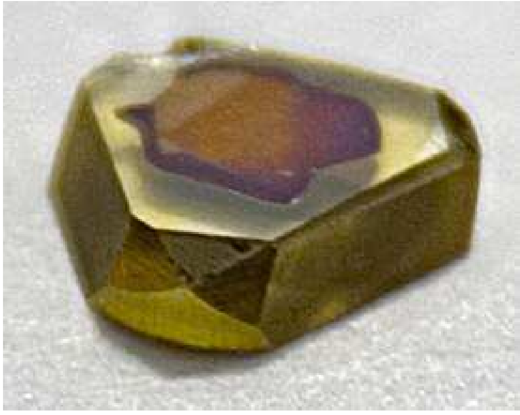

FIG. 9: Sample 219 which demonstrated avalanche-like behavior.

caused by charge gradient due to nitrogen content non-uniformity. Nitrogen defects, capturing free charge carriers, become charged. So in case on non-uniform distribution of these defects large electric fields can arise. These fields can produce avalanche electron multiplication. So a possibility exists to develop an avalanche diamond detector with help of controllable process of nitrogen non-uniformity creation. Such a detector would have greater sensitivity. It will not also require expensive charge-sensitive preamplifier due to internal avalanche amplification.

4. Conclusions

Three tested samples proved that monocrystalline diamonds, produced with split sphere method, are able to work as particle detectors despite their non-ideal crystal structure and high impurity content. Low cost of crystals produced with this method together with intrinsically high radiation hardness of the diamond can provide wide range of applications for these detectors in the field of particle physics, nuclear power engineering, industrial processing of radioactive waste and any other field requiring extreme radiation hardness and good sensitivity. Medical applications are also possible due to tissue-equivalence of the diamond (its atomic number is close to that of alive tissue). Monocrystalline nature may also permit to achieve (at more advanced stage of research) good energy resolution, which is impossible for polycrystalline CVD detectors.

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