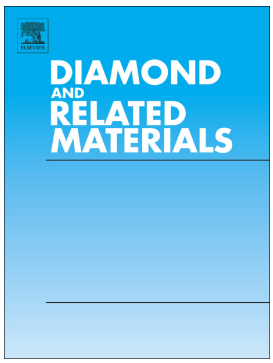

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High power density nuclear battery prototype based on diamond Schottky diodes

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Abstract

We report here for the first time a fabrication of betavoltaic battery prototype consisting of 200 single conversion cells based on Schottky barrier diamond diodes which have been vertically stacked with ~24% ^{63}Ni radioactive isotope. The maximum electrical output power of about 0.93 μW was obtained in total volume of 5x5x3.5 mm^3. We used the ion-beam assisted lift-off technique to obtain conversion cells of minimal thickness comparable with the characteristic penetration length of beta-particles emitted by ^{63}Ni isotope. The obtained value of 15 μm was limited by the mechanical strength of produced structures and process reliability. To check the performance of thin diamond based conversion cells we carried out IV-curves measurements at electron beam irradiation in SEM. We found that the sacrificial layer for the splitting of such thin conversion cell from HPHT diamond substrate did not cause a considerable degradation of device charge collection efficiency. As a result, the fabricated prototype provided the output power density of about 10 μW/cm^3, that is the best known value for nuclear batteries based on ^{63}Ni radioisotope. Moreover, the long half-life of ^{63}Ni isotope gives the battery specific energy of about 3300 mWh/g that is an order of magnitude higher than the typical value of commercial chemical cells.

Keywords: nuclear microbattery, betavoltaic, nickel-63 isotope, Schottky diode, synthetic diamond, ion beam implantation

1. Introduction

1.1. Betavoltaic batteries and applications

Nuclear power sources store energy in a radioactive isotope and convert it to electricity [1, 2]. Their key parameter is a huge specific energy that can be three orders of magnitude higher than the typical value for commercial chemical cells. It means that nuclear batteries can operate continuously without recharging or replacement for years or even decades – depending on the half-life of the radioisotope – even under extreme temperatures at which chemical batteries irrevocably fail. They offer unique advantages for use under conditions that render device maintenance difficult, impossible or life threatening.

Despite relatively low constant output power density a nuclear battery trickle charging some kind of electrical accumulator (secondary chemical cell or capacitor) can provide pulsed output power in the range of milliwatts to watts [3, 4]. It enables long-life power supply for a wide range of remote electronic systems like sensors, various analog and digital diagnostic microchips and wireless communication systems that can operate in space, desert, tundra, ice and undersea or without any maintenance during more than 30 years.

Other possible applications of nuclear batteries include implanted medical devices whose long lifespan can improve life quality of patients due to reducing healthcare costs and eliminating trauma associated with periodical invasive surgeries for maintenance. For example, cardiac pacemakers, defibrillator, cerebral neurostimulators, cochlear implants, intraocular implants and others implanted devices could be powered by small-size nuclear power sources [5-8].

1.2. Direct energy conversion in diamond diodes

Any nuclear battery consists of a radioisotope source and a converter and is characterized by its total efficiency η that can be separated into two portions:

$$\eta = \eta_{source}\eta_{conv}, \quad (1)$$

where η_{source} is the total power entering into the converter divided by the total power produced in the radioisotope source (efficiency of source using), η_{conv} is the total electrical power output of the battery divided by the total power entering the converter (conversion efficiency).

Utilizing the direct energy conversion by semiconductor converter is one of the most promising ways to create efficient nuclear battery [9, 10]. The principle of operation for such a device is similar to well-known photovoltaic solar cells. High-energy particles emitted by radioisotope nuclear decay (electrons in betavoltaic sources) interact with semiconductor material and create nonequilibrium charge carriers (electron-hole pairs – EHPs) that drift under the influence of diode junction internal electrical field.

Today multiple prototypes of betavoltaic microbattery based on different semiconductor materials such as silicon, silicon carbide and gallium nitride have been reported [10-16]. Compared to narrow bandgap materials, wide bandgap semiconductors are preferable for energy converter fabrication because the maximum conversion efficiency η_{conv}^{max} increases almost linearly with the bandgap [17]. Moreover, devices based on such materials can operate at elevated temperatures and usually are radiation resistant. This makes operation in harsh environment conditions possible and significantly reduces the rate of structure defects generation by radiation damage from radioisotope and outer sources like cosmic rays. Diamond possesses a wide bandgap (5.5 eV) and is an extremely radiation hard material [18], thus, diamond-based converters have the potential to be the most stable, long-lived and powerful among the other semiconductors.

Recently we reported for the first time a prototype of planar nuclear battery with the overall active area of about 15 cm^2 consisted in 130 single cells of Schottky barrier diamond diodes [19]. The fabricated battery provided up to 45 nW of output power under beta irradiation of 2 μm thick layer of low enriched ^{63}Ni that has been electrolytically deposited on 50 μm thick stable Ni foil. Due to the long lifetime of ^{63}Ni isotope, this unoptimized design already had the specific energy of about 100 mWh/g, comparable with the commercial chemical cells. However, since the total thickness of the diamond-based conversion cells was about 60 μm, the other important battery parameter – power density – was only about 0.3 $\mu W/cm^3$.

Poor volume characteristics of the battery was caused by non-optimized thickness of both conversion cells and radioactive source.

In this study we proposed to use ion-implantation assisted lift-off method [18] to split the CVD layer from the HPHT substrate in order to make super-thin diamond conversion cells. Applying this technique, the large array of 15 μm thick conversion cells based on Schottky barrier diamond diodes has been produced. After that we connected 200 thin diamond-based conversion cells in parallel by alternating cells of opposite polarity with 2 μm thick ^{63}Ni foil to obtain single vertical stack. The fabricated battery prototype with proposed 3D-architecture provided a total output electrical power of 0.93 μW and power density of about 3300 mWh/g that is an order of magnitude higher than our previous result.

2. Materials and methods

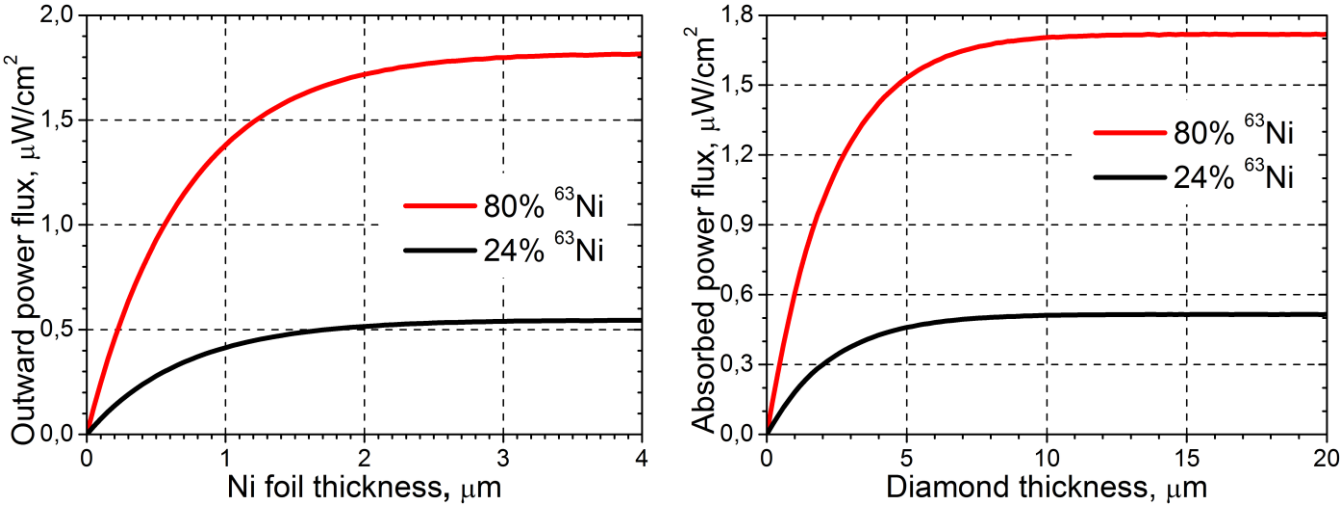
2.1. Battery volume optimization

The most important challenge in the development of any nuclear battery is to achieve the maximum possible output power density during its full lifetime. All long-lived isotopes with $T_{1/2}$ > 50 years have low activity and thus low initial power density. We proposed the multi-planar 3D-architecture of the battery and optimized its power density by thinning material layers down to optimal thickness and removing other parts that are unnecessary for electric power generation.

First of all, to improve battery power density we had to remove any inactive holder of isotope and produce beta-source with optimal thickness by minimizing the self-absorbance effect in the source itself. Most of the functioning beta-voltaic batteries prototypes described earlier contain a radioisotope in form of a thick substrate on which a thin layer of a radioisotope was deposited or thin film deposited directly on the converter cell surface. These approaches were unacceptable to us because they both lead to addition of some unused volume to our 3D-architecture: a substrate in the first case and some form of electrical connector like a wire or foil in the other case. We decided to use ^{63}Ni foil both as the radioactive source and the electrical connector.

The main role of the converter is to absorb the kinetic energy of beta particles and transform it into the electricity. For commonly used isotopes ^{3}H, ^{63}Ni and ^{147}Pm the decay electrons stopping path varies from about 1 to 50 μm depending on the converter material and geometry, but usually beta-voltaic batteries prototypes include at least 100 μm thick converters for technological reasons. Only the part of the converter in which the beta particles are absorbed is needed, and the remaining part limits the battery volume efficiency and, therefore, must be removed.

To determine the optimal thickness of the radioisotope foil and diamond converter we performed a Monte-Carlo simulation for the transmission of the betas from ^{63}Ni decay through the source into the converter body using the GEANT4 code [20] (see Fig. 1). The code takes into account the complete three-dimensional problem geometry, the continuous ^{63}Ni beta spectrum rather than the average beta energy and backscatter of beta particles from the source/converter interface. According the calculations, the outward power flux entering into a converter from one side surface of ^{63}Ni planar source becomes maximum at 2 μm thickness of isotope layer due to high self-absorption of decay electrons inside the source itself. Moreover, the total power absorbed by 1 cm^2 of the converter saturates at diamond thickness of about 10 μm. Thus, the thickness of the converter should be as close as possible to this value.



a) b)

Fig. 1. Dependencies of the electron power flux on the thickness of the radioactive Ni foil (a) and the power absorbed by 1 cm^2 of the diamond plate on its thickness (b). Percentage of ^{63}Ni refers to the

content of ^{63}Ni in the foil: 24% for nickel obtained from the high-flux nuclear reactor and 80% for centrifugally enriched ^{63}Ni.

2.2. Novel super-thin diamond converters

Diamond conversion cell operating principle is described in [19] and differs a little from the operation principle of a p-n silicon cell [9]. Generally, the p-n or p-i-n structure yields better results for betavoltaic appliances than Schottky barrier structure on the same material because it allows the collection of EHPs from much larger volume based on the doping profile and increases the cell voltage. However, since stable shallow n-type doping of diamond is still not possible, we decided to use the Schottky barrier structures on low-doped p-type diamond.

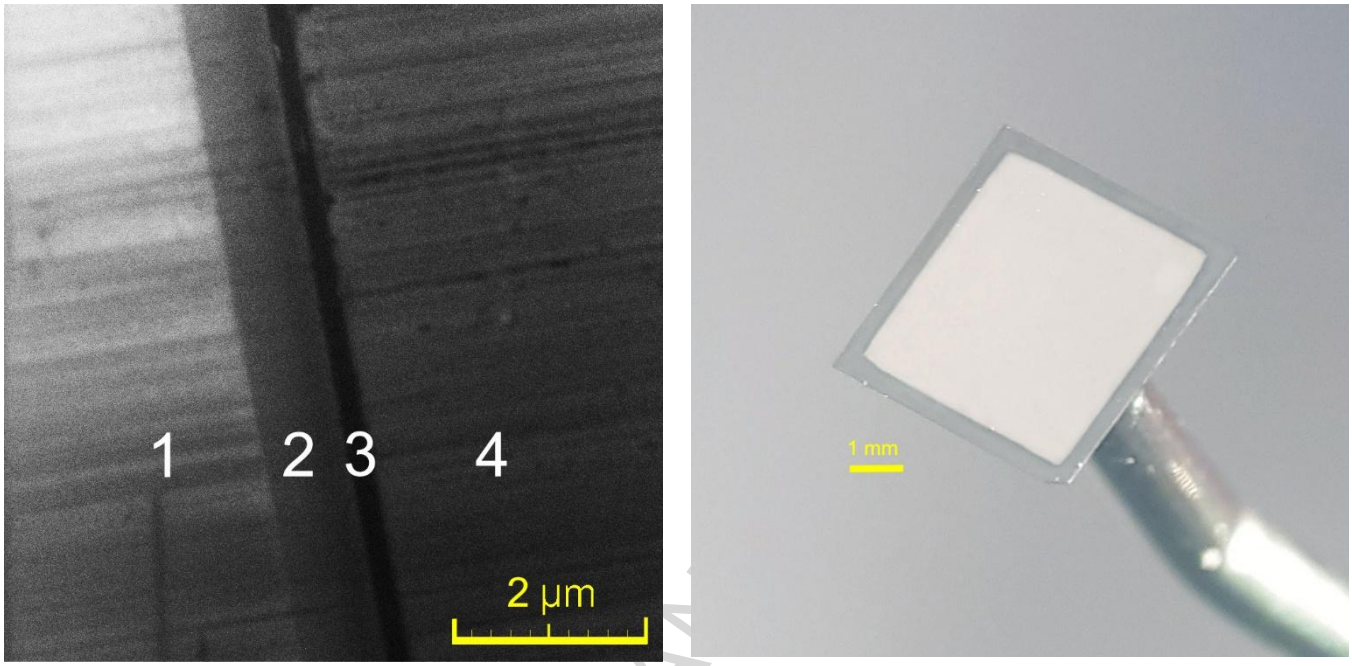
Making less than 100 μm thick diamond converters by mechanical polishing or plasma etching is a very complicated task. In this study we used the ion-implantation assisted lift-off method [21] to split the CVD layer from the HPHT substrate in order to make a large array of 15 μm thick conversion cells based on Schottky barrier diamond diodes.

We used the laser cut bulk synthetic boron-doped {001} diamond plates with a boron content up to 10^{18} cm^{-3} as p+ substrates. The initial crystals were grown by the temperature gradient technique under high pressure [22]. Substrates with low density of extended defects were selected by X-ray topography and UV-luminescence mapping [23]. All substrates were shaped by laser to 5×5 mm^2 500 μm thick plates and then polished mechanically.

In order to realize the lift-off technology, we implanted ~10^{16} cm^{-2} 450 keV He+ ions into a face of each plate using High Voltage Engineering Europa accelerator system (HVEE 500). The carbon atoms displacements (damage) profiles caused by ion implantation were calculated by TRIM code [24]. Implantation dose was chosen to ensure creation of ~100 nm layer with damage over the threshold for the ion-beam induced graphitization [25] at the maximum available ion energy of 450keV for the HVEE500.

The substrates then were annealed at 1400°C in vacuum to convert the buried highly damaged layer to sp2 phase and recover top diamond layer. 15 μm thick diamond epitaxial film with ~10^{15} cm^{-3} boron

content was grown by MP-CVD technique with tunable B_2H_6 gas addition. SEM analysis of the fabricated structure cross-section (see Fig. 2a) reveal the formation of graphitic-like layer at the predicted depth of 700 nm but with slightly higher thickness than was predicted by calculation.



a) b)

Fig. 2. (a) SEM image of of homoepitaxial CVD diamond layer cross-section (1) on thin part of HPHT diamond substrate (2) with thin He-implanted buried graphitized layer (3) for lift-off located between (2) and main part of the HPHT diamond substrate (4); (b) 15 µm thin Schottky barrier diamond diode caught by a vacuum tweezer.

Then the thin bilayer diamond plates were separated from the bulk crystal by electrochemical etching of the 100 nm thick graphitized ion-damaged layer. Finally, the Ti/Pt/Au ohmic contacts (positive electrodes) and thin Ni Schottky contacts (negative electrodes) have been deposited by magnetron sputtering at the opposite faces of bilayer diamond plates to make Schottky diodes [21] (Fig. 2b). We checked the doping of the drift CVD layer for one sample for each CVD process by capacitance-voltage profiling at reverse bias [26].

As a result, our energy conversion cell was a single-crystal diamond plate with metal contacts on the opposite sides (see Fig. 3). The plate consisted of two layers: a super thin (less than 1 µm) remains of

heavily doped p+ substrate and a 15 μm thick low doped p- functional drift layer. Details of thin Schottky diode structures production are described in [21].

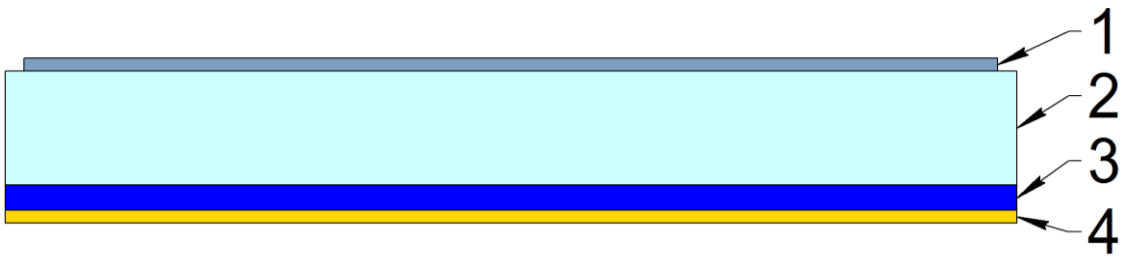


Fig. 3. A schematic view of a diamond conversion cell: Schottky contact (1), CVD p- drift diamond layer (2), p+ substrate (3), ohmic contact (4).

If several semiconductor converters are connected in parallel, the performance of the entire assembly is determined by the one having the least voltage. To ensure the best result we tested all manufactured diodes and selected the suitable ones by parameters: leakage current less than 100 pA at reverse 10 V; the rectification ratio more than 10^8 at 2 V bias; the turn-on voltage more than 1 V. The typical dark IV characteristic and other details on thin diamond Shottky barrier diodes are presented in [21]. After etching HPHT substrates were used for the next implantation-CVD-etching processes, that allowed us to manufacture 200 good diodes (cells) using only 20 reusable HPHT substrates.

2.3. Nickel-63 foil source

To reduce the needless volume of the radioactive source, we used the low enriched (~24%) ^{63}Ni in the form of 2 μm thick rolled foil. The manufacturing process was based on method presented in [27]. A solution of ^{63}Ni in hydrochloric acid, obtained after its neutron irradiation in nuclear reactor, was mixed with oxalic acid, resulting in the production of nickel oxalate. Nickel oxalate was converted to ^{63}Ni metallic powder by annealing in a hydrogen atmosphere. The powder was fused into an ingot. Then the ingot was rolled repeatedly until a thickness of 30 μm was reached. The foil was then placed in an envelope of polished stainless steel and rolled until a thickness of 2 μm was reached. Further thinning of the foil resulted in drastic drop in its quality and mechanical properties.

2.4. Battery prototype assembly

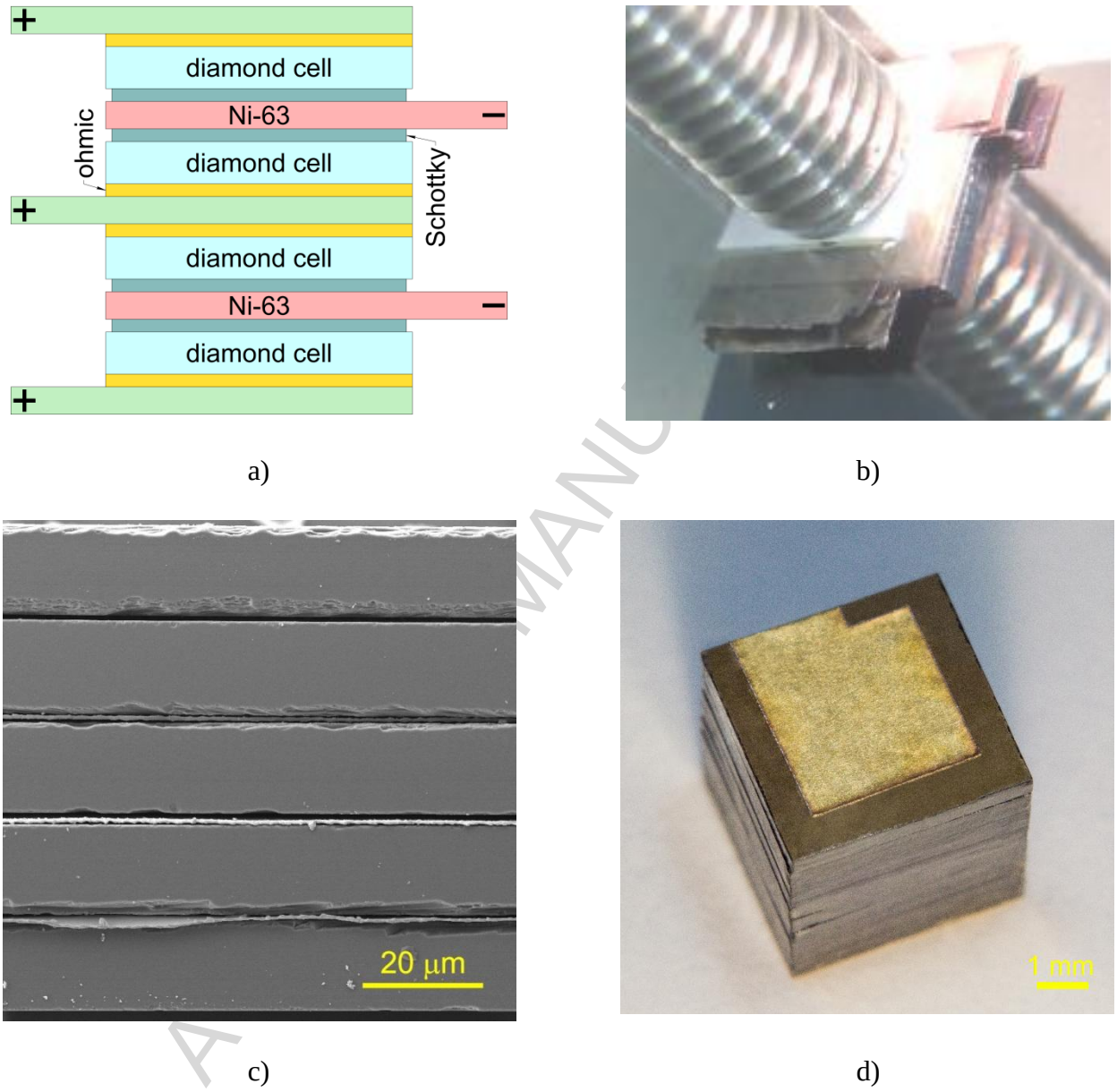
Our previous studies have shown that a single diamond conversion cell can generate about 1 V open circuit voltage [19], however, the current of a single cell is only about few nA. To power up a functional device one usually needs at least a current of 1 μA, so we connected all conversion cells in parallel. To minimize the volume of all battery elements we developed the multilayer 3D-architecture of the converting cells, radioisotope and electrical connectors [28], consisting of thin diamond cells connected in parallel by alternating cells of opposite polarity with 2 μm thick ^{63}Ni foil to obtain a single vertically stacked battery.

In our previous work [19] we used ultrasonically welded 40 μm golden wires to electrically connect individual cells in parallel which added at least 40 μm to every cell thickness. In the new 3D-architecture we placed stable nickel foil connectors between the ohmic contacts of adjacent cells. These connectors were rolled the same way ^{63}Ni source was and have the same thickness. A 200 nm Au layers were deposed on both sides of connectors to ensure the best electrical contact by a soft metal.

The nuclear battery prototype consisted of 200 diamond converter cells, 100 ^{63}Ni sources and 100 stable nickel connectors. We made 10 glued packs of 20 converters each, and then assembled the battery of them. Diamond cells were combined in pairs so that the Schottky contacts face each other, radioactive sources were located by one between the contacts so that their parts protrude from one side of the stack (see the schematics on Fig. 4a). The electrical connectors were placed between the pairs of cells so that their parts protrude from the other side of the stack. We assembled and glued each pack individually in the technological mandrel (see Fig. 4b). Then we bent the foil of the sources and connectors along the ends and glued the sides of the package. Each packages has size of 5×5 mm^2 and thickness about 0.35 mm, positive electrodes on three sides and negative electrode on one side. The laser-cut cross-section of a test package (with stable nickel instead of radioactive source) is show on Fig. 4c.

A set of 10 cell packs was stacked on the top of each other (see Fig. 4d), connected by electrodes on their sides in parallel the same way as individual cells were connected in packages and glued together by

epoxy. Sizes of the battery were 5×5×0.35 mm^3. The total activity of ^{63}Ni isotope (life-time ~100 years) used in our assembly was estimated as 0.55 Ci (20 GBq) with the total decay thermal power of 74 μW which is completely negligible for device heating.



a) b)

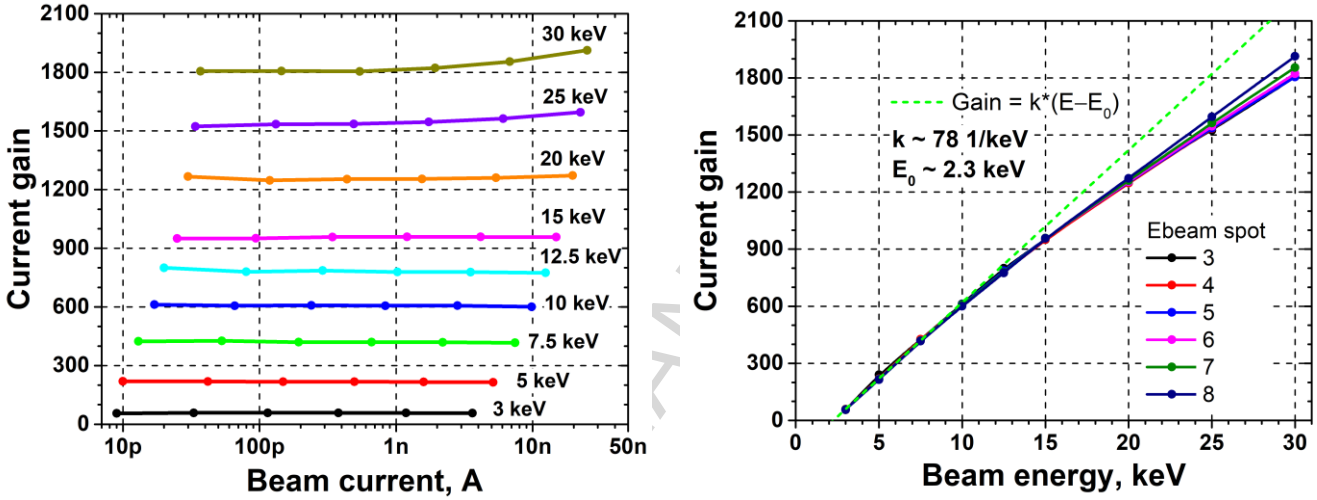
c) d)

Fig. 4. The cell package: scheme (a), assembly (b), SEM image of cross-section (c) and photo of assembled battery (d).

3. Experimental results and discussion

3.1. Single conversion cell characteristics

We tested each individual cell as betavoltaic converter employing electron beam induced current method (EBIC) [29] in Quanta 200 FEI SEM before assembling the battery. We measured the induced current gain values (number of EHPs generated by one electron of a primary beam and collected by a measuring system) in a short-circuit mode at beam energies up to 30 keV and beam currents up to 30 nA (see Fig. 5).



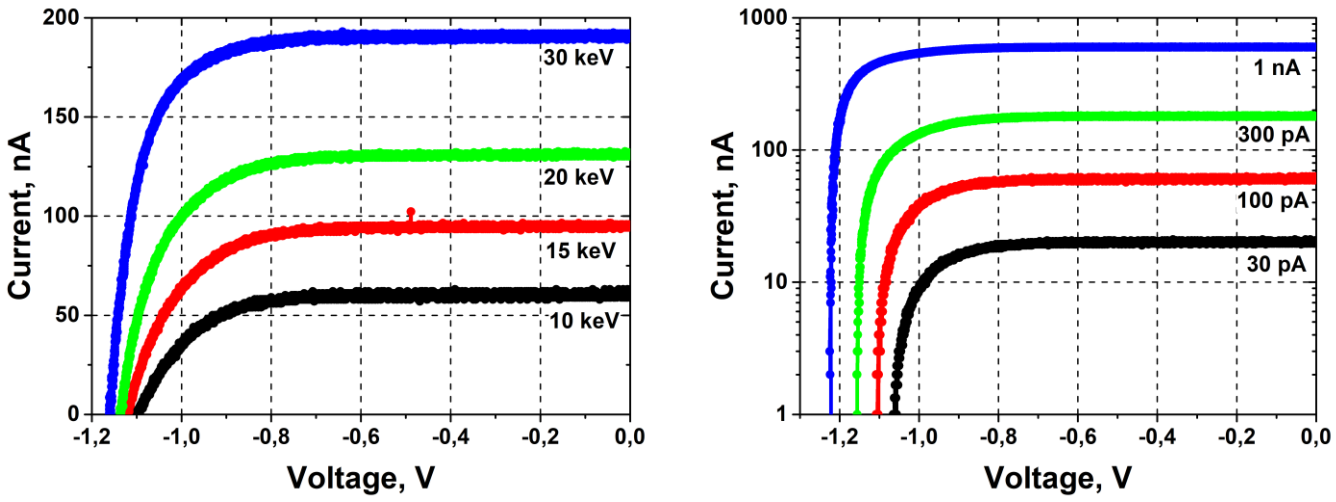
a) b)

Fig. 5. The induced current gain in a short circuit mode (zero bias voltage applied) vs the primary beam current (a) and its energy (b).

As shown on Fig. 5a, the gain remained constant for different power fluxes at the same beam energy, which means that the EDPs collection in the converters was not limited by a space charge up to 1 mW of the total beam power. Moreover, an increase of the beam energy up to 15 keV at a constant electrons flux results in a linear increase of the induced current in sample (Fig. 5b). Linear regression in this energy region gave us the mean energy for single EHP creation that equals to ~13 eV, which is consistent with the usually quoted value [30]. Above 15 keV the current gain increased as well, but with the decreasing slope because EHPs created deep inside the diamond recombine before reaching contacts. We observed a gain of about 1200 and less than 10% of EHPs loss due to recombination at the mean

energy of ^{63}Ni isotope beta-radiation (~17 keV). It means that the carriers diffusion length in the CVD diamond layer synthesized on the HPHT substrate after the ion implantation is of the order of several microns, which is not the best achieved value but is definitely enough to effectively collect EHPs generated by ^{63}Ni decay electrons.

The typical photovoltaic characteristics of the fabricated diamond-based conversion cells under electron beam irradiation are presented in Fig. 6. Thin diamond converters exhibit a significant betavoltaic effect under incident power fluxes close to possible values of real ^{63}Ni radioactive source. Because of the very low reverse leakage current for diamond diodes in dark mode the open-circuit voltage more than 1 V was achieved even for only 100 nW of SEM beam power. The charge collection efficiency in a short-circuit mode exceeded 90% at a beam energy less than 20 keV. It means that even at zero bias almost all EHPs generated by ^{63}Ni source will be collected. The filling factor typical value was 74-78%. The calculated cell conversion efficiency slightly changed with the beam energy from 4.5% at 10 keV to 6% at 30 keV due to the relative decrease of incident electrons backscattering and energy loss in metal Schottky contact.

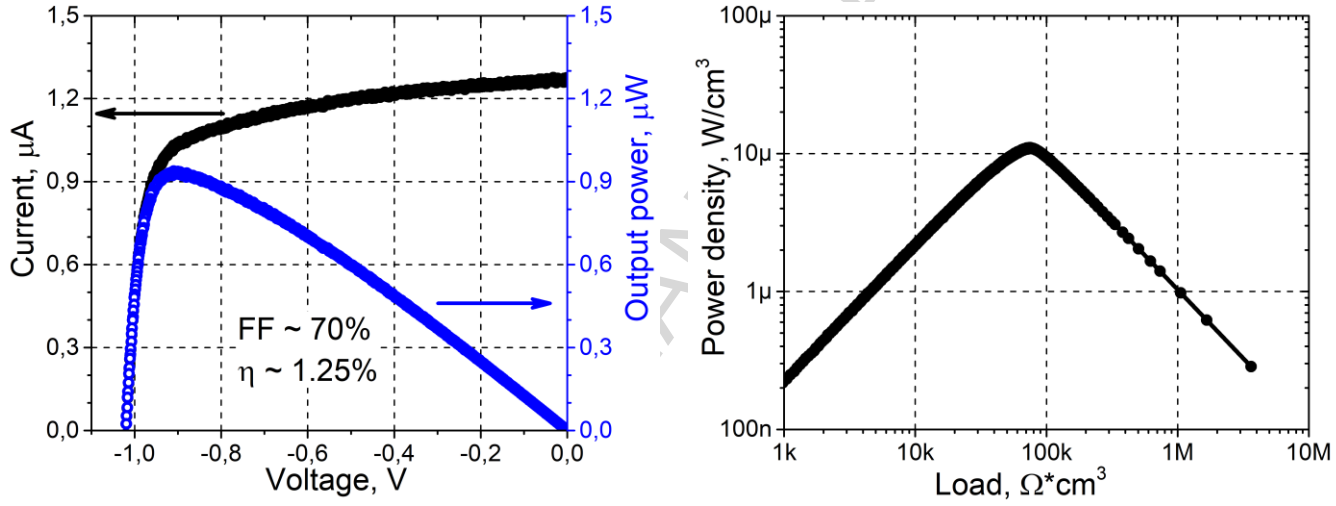


a) b)

Fig. 6. Typical photovoltaic curves for thin diamond conversion cell at electron beam irradiation: variations of primary beam energy (a) at constant current of 100 pA and primary beam current (b) at constant energy of 10 keV.

3.2. Battery prototype performance and possible applications

The current-voltage characteristic, output power vs voltage and power density as a function of the volume-normalized electrical load for the assembled ^{63}Ni battery prototype are presented in Fig. 7. The open-circuit voltage and the short-circuit current were 1.02 V and 1.27 µA, respectively The maximum output power of about 0.93 µW was obtained at 0.92 V. The optimal electrical load was ~70 kΩ·cm^3. The output power density was 10 µW/cm^3 and the specific energy was calculated to be 3300 mWh/g that is an order of magnitude higher than our previous result [19]. Taken into account the total decay thermal power of ^{63}Ni amount used in the prototype the overall battery efficiency of about 1.25% was estimated.



a) b)

Fig. 7. Current-voltage, output power-voltage characteristics (a) and the power density vs volume-normalized electrical load (b) of 3D-architecture diamond betavoltaic battery of parallel-connected 200 diamond Schottky barrier diodes with ~24% ^{63}Ni radioactive source.

Battery with constant output of 10 µW and size of 1 cm^3 is enough to power some pacemaker models or to use as on-chip power supply. For example, Azure™ W1SR01 pacemaker by Medtronic has total volume of 12 cm^3, power consumption about 9 µW (at 100% pacing) and energy capacity less than 1400 mWh [31]. To accumulate enough electrical power for short-term powerful energy consumption that can be used to send electromagnetic signals or even trigger some actuators one must connect a low leakage current charge storage device with high enough input electrical resistance to the battery. But for

many applications higher output power and power density are needed. Since our technology can be used to create the nuclear battery with any radioactive nickel foil, the most direct way to increase the power density is to increase the ^{63}Ni isotope enrichment up to 80% or more by the centrifugal separation of isotopes.

We propose two ways to further optimize the ^{63}Ni-based battery by increasing the total efficiency of the energy conversion. Each diamond cell loses about 60% of all potentially convertible energy due to low potential barrier (Schottky junction in our case) height. It limits the open-circuit voltage of diamond converters and, therefore, its electrical power. Improvement of the metal-diamond interface preparation may allow us to increase the battery voltage about two times [32]. Moreover recent advances in active n-type diamond synthesis [33] present an opportunity to develop a diamond converter with p-i-n junction that will bring the conversion efficiency close to the theoretical limit by further increasing the open-circuit voltage more than 3 V and removing the metal Schottky contact layer.

Another optimization of the battery structure can be done by 3D structuring the surface of each diamond cell for example by the deep reactive ion etching [34]. But to fabricate a compact battery based on such cells we have to develop a new way to electrically connect all battery elements in minimal volume and a technology for ^{63}Ni deposition onto such increased surface.

4. Conclusions

We fabricated for the first time a prototype of 3D-architecture diamond betavoltaic battery with ^{63}Ni radioactive isotope and measured its electrical characteristics. The size of the battery was 5×5×3.5 mm^3. The maximum output power of 0.93 μW was obtained at about 0.9 V and the normalized optimal electrical load of 70 kΩ*cm^3. The unique well-optimized 3D-architecture provided the output power density of about 10 μW/cm^3 and the specific energy of 3300 mWh/g, which are the best specific power parameters for the known nuclear microbatteries based on low-enriched (~24%) ^{63}Ni radioisotope. Our technology can be used without changes to create a nuclear microbattery on highly enriched ^{63}Ni radioisotope that will have the specific power of 40-50 μW/cm^3.

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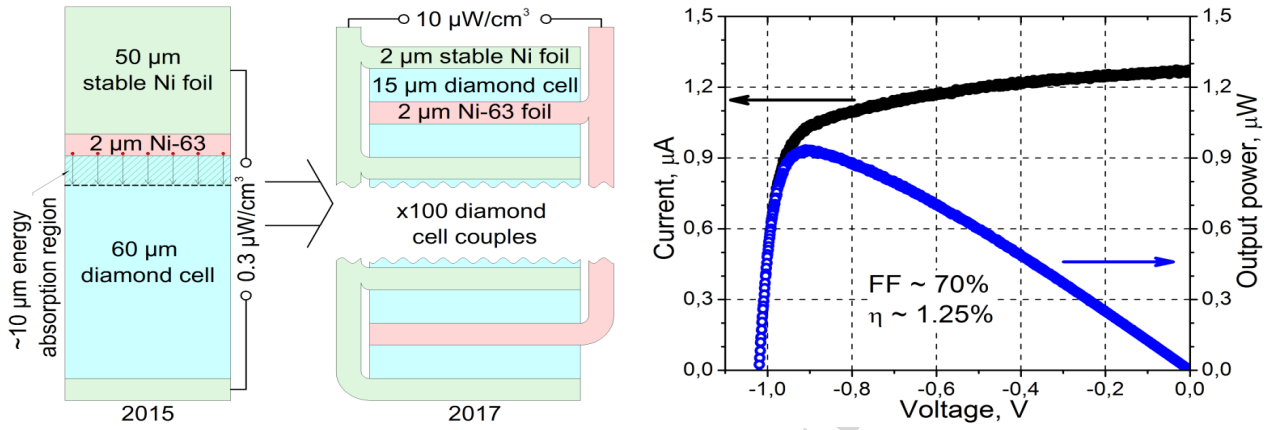

Graphical abstract

Diamond and Related Materials

Highlights

- We successfully used ion-beam assisted lift-off technique to fabricate a set of energy conversion cells based on 15 μm thick diamond Schottky barrier diodes
- The energy conversion efficiency (~5-6 %) of produced conversion cells was limited b relatively low open-circuit voltage (~1.1 V for a typical cell), although high charg collection efficiency (>90 %) was demonstrated.
- Compact (~90 mm^3) and light-weight (~0.35 g) nuclear battery prototype with a total output electrical power of 0.93 μW was developed and fabricated by using of 200 parallel connected diamond-based conversion cells combined with low-enriched (~24%) ^{63}Ni radioisotope foil.
- The battery power density of 10 $\mu W/cm^3$ and specific energy of 3300 mWh/g were achieved due to conversion cell thickness decreasing by removing of diamond substrate.