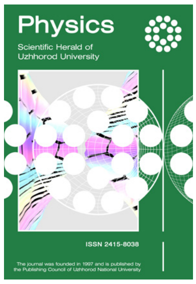
Scientific Herald of Uzhhorod University
Series “Physics”

Journal homepage: https://physics.uz.ua/en
Issue 50, 25-30
Received: 14.10.2021. Revised: 15.11.2021. Accepted: 19.12.2021



UDC 621.762.4.045; 621.921.34
PACS 62.20.−x.81.70.Bt
DOI: 10.24144/2415-8038.2021.50.25-30

Dependence of the Static Strength of Type Ib Single Crystal Diamonds with Octahedral Habit on Their Size

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Abstract

Relevance. Scientific and technological progress in diamond drilling is aimed at increasing productivity and reducing the cost of the process. One of the crucial tasks of the last fifty years is to re-equip geological exploration and mining operations with a more efficient tool. This has led to new trends such as the reinforcement of the cutting edge through optimal processing methods, the use of hard alloys based on borides, nitrides and carbides, the use of natural diamond raw materials, and since 1956, the use of synthetic diamonds. Progress in the production of synthetic diamonds allowed making a breakthrough from fine-grained industrial powders consisting of type IIb crystals with a high content of solvent metal inclusions to non-magnetic type Ib crystals of increased strength up to 2 mm in size. Since the main load during operation is directed along the normal to the working surface of the installed crystal, it was decided to use crystals of octahedral habit, which is associated with the anisotropy of the strength properties of diamond.

Purpose. Determination of suitability for the use of non-magnetic type Ib single crystals of octahedral habit as materials for the production of drill bits.

Methods. The method for determination of the static strength for single-crystal substances at a constant load rate is used.

Results. The process of diamond destruction under static load is experimentally studied. It was established that crystals are susceptible to brittle fracture and the decrease in the size of fragments is reduced in proportion to the fracture force. The dependence of the static strength of type Ib single crystals of octahedral habit on the length of the side face is obtained.

Conclusions. Crystals with an edge size greater than 1200 μm have a lower static strength value compared to smaller crystals, the edge size of which varies between 1000...1100 μm. Large single crystals of type Ib synthetic diamond of octahedral μm with size 1100/1600 can be used to make drilling and dressing tools

Keywords: type Ib synthetic diamond, diamond drilling tool, geological exploration, physical and mechanical properties, HP-HT temperature gradient method

Suggested Citation:
Tsysar MO, Zakora AP, Babak AM, Ivakhnenko SO, Zanevsky OO. Dependence of the static strength of type Ib single crystal diamonds with octahedral habit on their size. *Scientific Herald of Uzhhorod University. Series “Physics”.* 2021;(50):25-30.

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Introduction

In the process of manufacturing tools based on poly- or single-crystal diamonds, it is important to know to what extent the initial properties of the crystals will ensure the operational characteristics of the tool. Notably, of all types of diamond tools, the drill bit operates in the most difficult conditions due to the heterogeneity of rocks, the difference in loads during operation, and the lack of operational monitoring of the condition of the cutting part. The use of low-quality diamond raw materials in drill bits leads to rapid wear of their diamond-containing matrix, which, in turn, can lead to an increase in tripping operations during drilling and a decrease in its efficiency. That is why the right choice of high-quality raw materials for use in drilling tools is so important.

160 years have passed since the invention of the first diamond drill bit in 1862. According to one version, George Leschot requested 100 carats of diamonds, and there are also two other versions of what was used for this: round ballas diamonds or black industrial diamonds of the Carbonado variety [1; 2]. The single crystals of the diamond used for the production of the drill bit weighed 0.5...2 carats. Such drill bits were used until the mid-1950s.

The new trend was the use of small diamonds, this innovation was aimed at reducing the cost of production and simplifying the design. Crystals and their fragments were arranged at the cutting face in one or more layers, depending on the size. The main achievement of this design can be considered the absence of any arrangement scheme and, accordingly, jewellery work on inlaid diamonds in the drill bit. Thus, the following diamonds were used for the production of drilling bits: single-layer 40...60 pcs./carat, multi-layer 60...120 pcs./carat impregnated with a volume distribution of 120...500 pcs./carat [3]. The use of diamond industry waste in the form of powders allowed achieving the desired result – an effective tool with a low cost. After the discovery of kimberlite pipes in Yakutia in 1954, heavy-duty powders made from colourless diamonds were used in the diamond drilling tools manufactured in the USSR [2].

Therewith, the area of manufacturing and introduction of synthetic diamonds was developed. In 1939, the study by O.I. Leipunskiy was published in the journal "Successes of chemistry", which contained a diagram of the thermal stability of a diamond. In 1953, the first batch of 2,000 carats was produced in the Institute of Superhard Materials using this technology [4]. In the second half of the 1960s, under the leadership of V.M. Bakul, a whole series of tools was developed using synthetic diamonds for industry, including in drill bits. During this period, a large number of works were carried out in the following areas: introduction of polycrystalline diamond inserts [5], reinforced hybrid ACTM [6], reinforced superhard materials of the Slavutich type [7]. In cooperation with foreign partners, technologies for using CVD diamonds were introduced [8].

Recently, there has been an active transition to the use of large artificial diamonds for the manufacture of rock-breaking tools, which helps to increase stability in operation. Synthetic diamonds of the appropriate quality not considerably but have a lower cost than natural ones of similar sizes. In terms of physical and mechanical strength, hardness and temperature resistance, such crystals are not inferior to natural ones. Therewith, synthetic single crystals have clear edges of the octahedron, or rhombic dodecahedron, which, for its part, provides a considerably higher cutting capacity during drilling than that of technical natural diamond raw materials.

This paper presents a study of the strength of large single crystals of type Ib diamond with octahedral habit with a size of 1.5...2 mm, obtained by the HP-HT temperature gradient, and which are planned to be used in the future for the manufacture of diamond dressing and rock-breaking tools.

Materials and Methods

As a rule, single-crystal diamonds of natural or synthetic origin are far from simple shapes or series of the cube-octahedron-rhombic dodecahedron type in their morphology. Thus, rhombic dodecahedron face-blunting edges can be present in crystals of octahedral habit, along with vertex-blunting cube faces. Primary analysis of the morphology of synthetic diamond crystals is carried out using optical devices, the resolution of which is selected depending on the size of the crystals under study.

The experimental batch consisted of 166 crystals. The crystals had an octahedral habit, with defects of this type: blunted vertices, blunted or double edges, growth steps, and surface irregularities associated with the synthesis method (Fig. 1). The initial inspection was carried out using a Skydust biological microscope XSP-1406 with a maximum magnification of 1200, equipped with a 5MP digital set-top box.

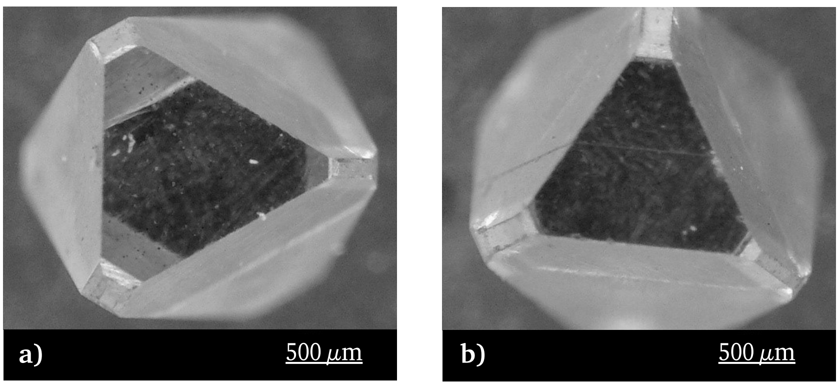

Figure 1. Image of type Ib octahedral habitus crystals from the experimental batch with an average edge length of a) 1.22 mm b) 1.36 mm

The geometric dimensions of the crystals in the experimental batch were measured according to the enterprise standard. Thus, the length of the edges was measured with a digital calliper manufactured by UKS, DCzag81520773, since the specified device complies with regulatory documentation and has a measurement accuracy of up to the second decimal place, and its error varies within ±0.01 mm [9]. To avoid the effects of hand tremor and unexpected distortion of measurements, which is associated with the skew of the crystal during the measurement process, a clamp with flat sponges was used. To avoid damage to the crystals – the appearance of cracks on the sides, an additional layer of technical velvet [10] was placed between the steel sponges and the prototype with sections of the corresponding rectangular shape.

Static strength was chosen as an evaluation criterion for the physical and mechanical properties of diamond powders in accordance with GOST 9206-80. Static strength is defined as the maximum load at which partial or complete destruction of the diamond grain occurs. To study the static strength of grinding powders, it is recommended to use the RMU-05-1 breaking machine, equipped with a reverse for obtaining compressive forces and a binocular microscope. The publications also recommend using DDA-33mp [11] as an automated alternative. Notably, the maximum static strength of 100/80 powders varies in the range of 245 N or less [12].

To determine the required fracture forces based on materials from several sources [11; 13; 14], the ultimate strength was estimated depending on the crystallographic areas and type of load. The theoretical shear strength limit according to Frenkel's theory [11] for the crystallographic area {111} is 120 hPa. According to the research data [15], the strength of crystals considerably depends on their size and perfection of structure. Thus, for octahedral habitus crystals with a size of 2.5 mm, the compressive strength limit is 8.9 hPa. Thus, based on theoretical information and research results, it becomes obvious that the use of RMU-05-1 and DDA-33MP is impractical since they cannot give the necessary compressive force. It was decided to conduct research on the FP-10 installation [16], with a maximum test force of 10,000 N, an error of ±10 N.

The FP-10 unit is not intended for studying samples of this type, and there are no appropriate means to ensure coaxial compression. According to this, there was a need to design and manufacture a special manipulator (Fig. 2). The design of the manipulator, due to the ball joint in the upper part, provides coaxial compression even if there is a skew between the base plates.

Figure 2. Manipulator equipped with carbide supports

Installing the sample in such a manipulator is carried out in several steps. Firstly, a layer of solidol is applied to the surface of the lower support located in the lower screw. Then a small piece of aluminum foil [16] of round or square shape with a linear size of 5 mm is laid out on this layer. In addition, a layer of solidol is applied to the foil, on which the studied single crystal is fixed. The single crystal is laid out with the largest side to the lower support using an auxiliary optical device, namely MAGNIFIER 81007-B glasses with X4 magnifying glass [17]. After that, the authors proceed to the preparation of the upper support, which is mounted in the upper screw. In the centre of the working plane of the upper support, a layer of solidol is applied, on top of which a piece of aluminium foil [16] of a round or square shape with a linear size of 5 mm is installed. The lower and upper screws are mounted in the manipulator body. Using the upper movable piston of the clamp, the upper support is mixed until it touches the surface of the single crystal, and the movement of the upper carbide support using the viewing window is controlled.

Results and Discussion

As noted in the methodological part, the initial examination was carried out using an optical microscope. The review resulted in a number of images (Fig. 1), based on which the following conclusions were made: there is no mechanical damage on the crystals, the crystals have shape and surface defects. No cracks were found on any of the crystals, which could have occurred as a result of their extraction from the melt-solvent matrix. The blunting of the vertices associated with the presence of the cube's habitus shape was taken as a minor defect since the width of the blunting of the vertex did not exceed 3...5% of the edge length. The blunting of the edges or their visual doubling is caused by the presence of the habitus shape of the dodecahedron, which in its width did not exceed 0.5...1% of the edge length and in some cases amounted to 1...1.5 microns. This defect also cannot considerably affect the static strength values of single crystals.

The influence of growth steps and surface defects could affect the plane-parallelism of the faces, thereby causing a skew during loading and causing an error in measuring the static strength value. To measure this value, a digital multi-turn indicator with an accuracy of 0.0001 mm Etopoo micrometre RS232 was used between the support and pressure faces. Studies have shown that the change in the value of plane parallelism associated with growth features and defects on the surface of the faces is not considerable and does not affect it, the crystals did not have considerable height differences at any point, this difference was within the measurement error of the research equipment, so the influence of this type of defects on the static strength value was also not considered.

In the next stage, according to the regulatory documentation [18], 5...10% was selected from the batch, which is 10 pieces in the case of the study. Geometric dimensions, namely the length of the edge, even for one sample could vary within 0.6...2.1 mm, so it was decided to take the average value for five measurements as the calculated value. The value of the edge size of the Octahedron was calculated using the formula (1):

$$a = \frac{a_1 + a_2 + a_3 + a_4 + a_5}{5} \quad (1)$$

where a_1, a_2, a_3, a_4, a_5 – measurement values of the octahedron edge.

The fracture pattern of all samples of the selected group was brittle. Complete destruction of a single crystal into small fragments occurs almost instantly. Notably, for crystals that collapse under a load of 2,000...10,000 N, the size of the fragments decreases (Fig. 3).

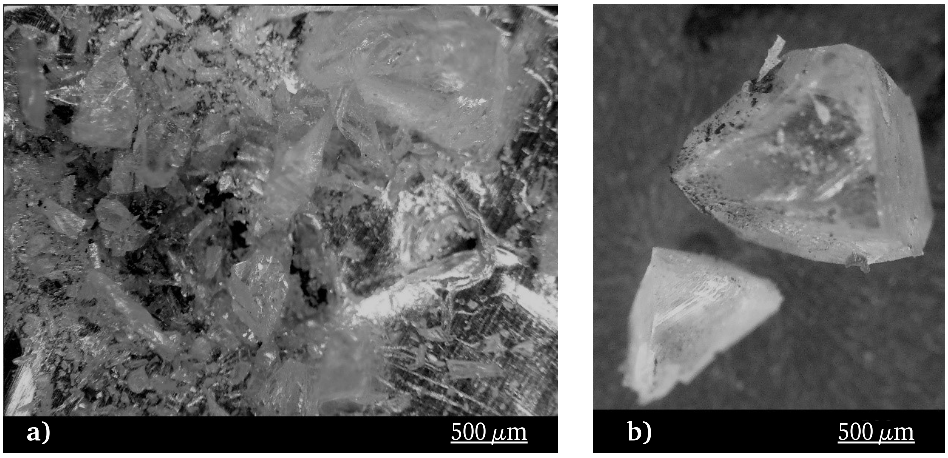

Figure 3. Artificial diamonds destroyed as a result of an experiment to determine the static strength a) 2150 N, b) 1850 N

Static strength according to DSTU 3292-95 is the force at which the crystal is destroyed. The values of critical load at which experimental type Ib single crystals of octahedral habit are destroyed are shown in Table 1.

Table 1. Results of tests to determine static strength

No.	Edge length a, mm	Force, N	Fracture pattern
1	1.02	5,000	Brittle, completely destroyed
2	1.03	5,150	Brittle, completely destroyed
3	1.06	2,150	Brittle, split in half
4	1.10	5,100	Brittle, completely destroyed
5	1.19	4,100	Brittle, completely destroyed
6	1.22	1,850	Brittle, completely destroyed
7	1.36	3,500	Brittle, completely destroyed
8	1.48	1,850	Brittle, completely destroyed
9	1.50	4,400	Brittle, completely destroyed
10	1.67	3,950	Brittle, completely destroyed

Based on the data in Table 1, the graph on the distribution of the critical compressive load or static strength from the size of a single crystal was designed (Fig. 4). Evidently from Figure 4, the data spread is significant. The crystals that showed the greatest breaking force have the side face length in the range of 1...1.1 mm, and the static strength decreases with increasing size. However, even in the range of 1...1.1 mm, there is a crystal with low mechanical properties, which could not be explained by conventional examination using optical devices. Therefore, for type Ib crystals of the same habit, an additional method of quality control is required, which would allow evaluating the quality of crystals by a non-destructive method and distributing such crystals into groups for premium tools and second-class tools.

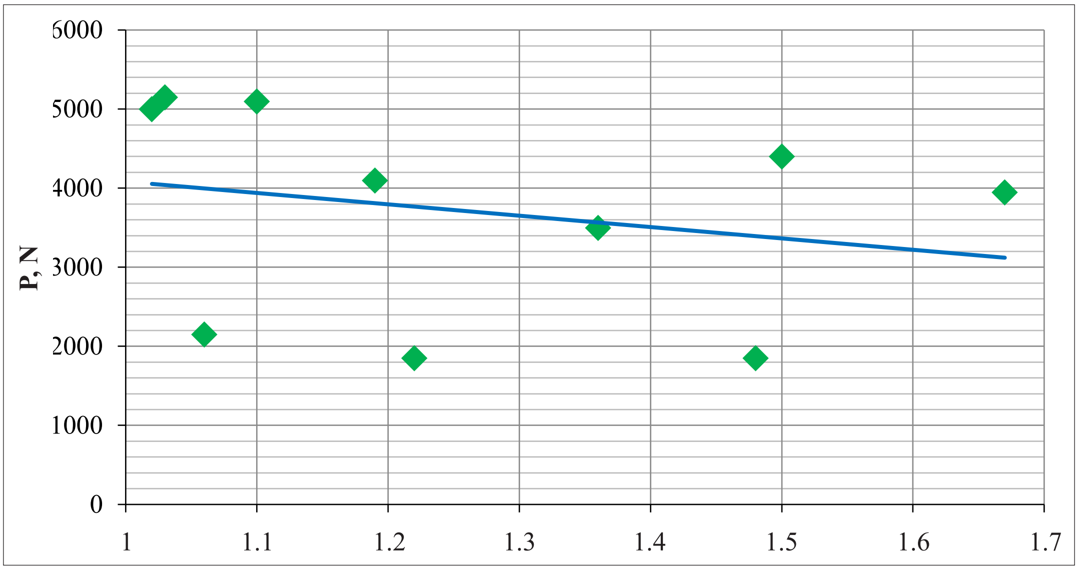

Figure 4. Dependence of static strength on the edge length of type Ib diamond single crystals

Conclusions

Summarising the above, it is worth noting that all large single crystals of type Ib synthetic diamond with octahedral habit size 1100/1600 can be used for the manufacture of drilling and dressing tools since they have high static compressive strength. However, experiments have shown that crystals obtained by the HP-HT method have strong differences in the value of static strength even for crystals of the same size. It has also been experimentally demonstrated that in the original crystals without additional thermal or chemical treatment, the static strength begins to decrease with increasing crystal size. That is, crystals of 1200 μm or more already have a lower value of the critical fracture load than crystals linear size of which is in the range of 1000...1100 μm. The issue of additional quality control remains open, since type Ib crystals differ considerably in their physical properties and chemical composition from typical diamond powders currently used in drilling and cutting tools.

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Залежність статичної міцності крупних монокристалів алмазу типу Ib октаедричного габітусу від їхнього розміру

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Анотація

Актуальність. Науково-технічний прогрес в алмазному бурінні спрямовано на підвищення продуктивності та зменшення вартості процесу. Одним з актуальних завдань останні п'ятдесят років є переозброєння геологорозвідувальних і видобувних робіт ефективнішим інструментом. У зв'язку з чим були відкриті нові напрями, такі як зміцнення ріжучої кромки за рахунок оптимальних методів обробки, використання твердих сплавів на основі боридів, нітридів і карбідів, використання природної алмазної сировини, а з 1956 використання синтетичних алмазів. Прогрес у виробництві синтетичних алмазів дозволив зробити ривок від дрібнозернистих технічних порошків, що складаються з кристалів типу IIb, з великим вмістом включень металу-розчинника до немагнітних кристалів типу Ib підвищеної міцності розміром до 2 мм. Оскільки в процесі експлуатації основне навантаження спрямоване по нормалі до робочої поверхні встановленого кристала було прийнято рішення використовувати кристали саме октаедричного габітусу, що пов'язано з анізотропією властивостей алмазу міцності.

Мета. Визначення придатності у використанні немагнітних монокристалів октаедричного габітусу типу Ib як сировини для виробництва бурових коронок.

Методи. Використано метод визначення статичної міцності для монокристалічних речовин за сталої швидкості навантаження.

Результати. Експериментально досліджено процес руйнування алмазів при статичному навантаженні. З'ясовано, що кристали руйнуються крихко і зменшення розмірів уламків зменшується пропорційно зусиллю руйнування. Отримано залежність статичної міцності для монокристалів октаедричного габітусу типу Ib від довжини бічної грані.

Висновки. Кристали з розміром ребра більшим за 1200 мкм, мають більш низьке значення статичної міцності, порівняно з кристалами меншого розміру, розмір ребра котрих змінюється в межах 1000...1100 мкм. Крупні монокристали синтетичного алмазу типу Ib октаедричного габітусу розміром 1100/1600 можна використовувати для виготовлення бурового та правлячого інструменту

Ключові слова: синтетичний алмаз типу Ib, алмазний буровий інструмент, геологорозвідувальні роботи, фізико-механічні властивості, метод HP-HT температурного градієнта