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(54) CUBIC BORON NITRIDE POLYCRYSTAL, CUTTING TOOL, WEAR-RESISTANT TOOL, GRINDING TOOL, AND METHOD OF PRODUCING CUBIC BORON NITRIDE POLYCRYSTAL

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(57) ABSTRACT

A cubic boron nitride polycrystal includes cubic boron nitride, the cubic boron nitride having an average grain size of not more than 150 nm, a ratio b/a being not more than 0.085 in measurement of Knoop hardness at 23° C.±5° C. under a test load of 4.9 N, the ratio b/a being a ratio between a length a of a longer diagonal line and a length b of a shorter diagonal line of a Knoop indentation.

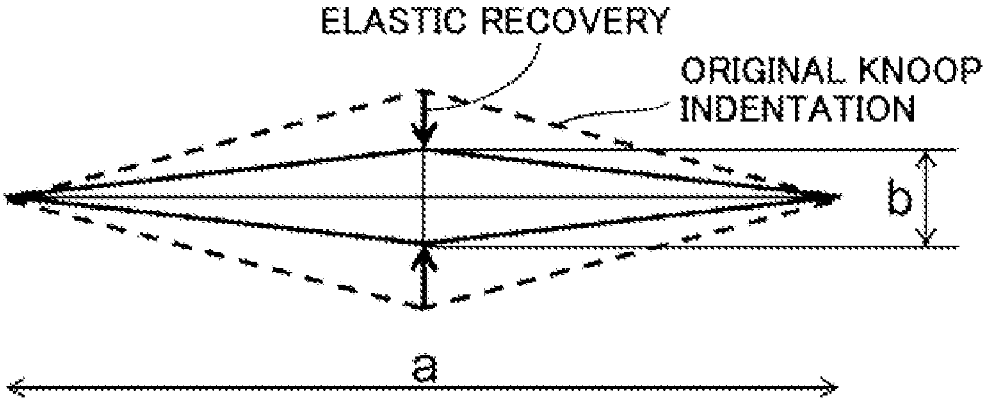
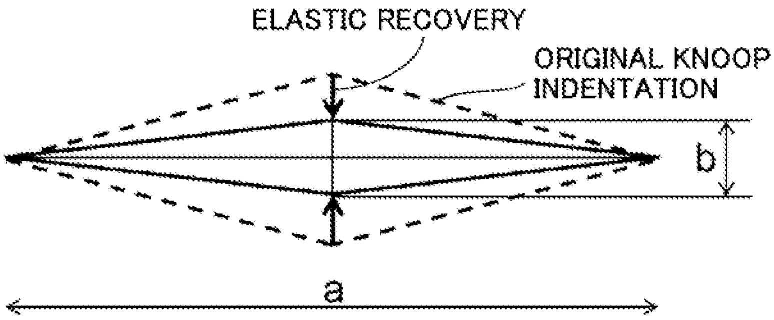


FIG.1



CUBIC BORON NITRIDE POLYCRYSTAL, CUTTING TOOL, WEAR-RESISTANT TOOL, GRINDING TOOL, AND METHOD OF PRODUCING CUBIC BORON NITRIDE POLYCRYSTAL

BACKGROUND OF THE INVENTION

[0001] 1. Field of the Invention

[0002] The present invention relates to a cubic boron nitride polycrystal, a cutting tool, a wear-resistant tool, a grinding tool, and a method of producing the cubic boron nitride polycrystal, in particular, a cubic boron nitride polycrystal, a cutting tool, a wear-resistant tool, a grinding tool, and a method of producing the cubic boron nitride polycrystal, which are useful as cutting tool, wear-resistant tool and grinding tool for an iron-based material.

[0003] 2. Description of the Background Art

[0004] Cubic boron nitride (hereinafter, also referred to as "cBN") has hardness second to diamond and is also excellent in thermal stability and chemical stability. Further, cubic boron nitride is more stable with respect to an iron-based material than diamond, so that a cBN sintered compact has been used as a working tool for the iron-based material.

[0005] However, the cBN sintered compact includes about 10 to 40 volume % of a binder, and this binder causes reduction of the strength, heat resistance, and thermal diffusion property of the sintered compact. Therefore, particularly when cutting an iron-based material at a high speed, thermal load becomes large and the cutting edge is likely to be chipped and cracked, resulting in a short life of the tool.

[0006] As a method of solving this problem, there is a method of producing a cBN sintered compact using a catalyst without using a binder. In this method, reaction sintering is performed using hexagonal boron nitride (hBN) as a raw material and using magnesium boron nitride (Mg_3BN_3) or the like as a catalyst. The cBN sintered compact obtained by this method does not include a binder, so that cBN grains are bonded to each other strongly and thermal conductivity becomes high. Therefore, the cBN sintered compact is used for a heat sink material, a TAB (Tape Automated Bonding) bonding tool, or the like. However, because a small amount of the catalyst remains in the sintered compact, a fine crack is likely to be caused under application of heat due to a difference in thermal expansion between the catalyst and the cBN and the cBN sintered compact is therefore not suitable for a cutting tool. Moreover, because the grain size is large, specifically, about 10 μm, the thermal conductivity is high but the strength is weak and the cBN sintered compact is therefore incapable of applications for cutting involving a large load or the like.

[0007] On the other hand, a cBN sintered compact can be also obtained by directly converting normal pressure type BN (boron nitride) such as hBN into cBN without using a catalyst under ultra-high pressure and high temperature and by sintering it at the same time (direct conversion sintering method). For example, each of Japanese Patent Laying-Open No. 47-034099 and Japanese Patent Laying-Open No. 03-159964 describes a method of converting hBN into cBN under ultra-high pressure and high temperature so as to obtain a cBN sintered compact. Moreover, there is a method of obtaining a cBN sintered compact using pyrolytic boron nitride (pBN) as a raw material. This type of method is illustrated in, for example, Japanese Patent Laying-Open No. 54-033510 or Japanese Patent Laying-Open No. 08-047801. In this method, conditions such as 7 GPa and not less than 2100° C. are required.

[0008] Each of Japanese Examined Patent Publication No. 49-027518 and Japanese Patent Laying-Open No. 11-246271 describes a method of obtaining a cBN sintered compact under conditions less strict than the above-described conditions.

SUMMARY OF THE INVENTION

[0009] Japanese Examined Patent Publication No. 49-027518 discloses a method of obtaining a cBN sintered compact under conditions such as a pressure of 6 GPa and 1100° C. In this method, the grains of hBN, which is a raw material, are of not more than 3 μm, so that the hBN includes several mass % of boron oxide impurity and adsorption gas. Accordingly, sintering insufficiently proceeds due to influence of the impurity and adsorption gas, and hardness, strength, and heat resistance become low due to the inclusion of oxide, with the result that the cBN sintered compact cannot be used as a cutting tool, a wear-resistant tool, and the like.

[0010] In order to solve the problem, Japanese Patent Laying-Open No. 11-246271 discloses a synthesis method performed using a low crystalline hexagonal boron nitride as a raw material under conditions of 6 to 7 GPa and 1550 to 2100° C. Moreover, it is disclosed that the cBN polycrystal synthesized by this method has a crystal grain size of about 0.1 to 1 μm.

[0011] However, when the sintering temperature is made low in order to obtain a cBN polycrystal having a small grain size useful for finish machining, precision processing, and the like, sinterability becomes low, which leads to low strength of the polycrystal. Furthermore, a small grain size leads to low toughness, with the result that a tool is readily chipped, disadvantageously.

[0012] In view of the above problem, it is an object to provide a tough cubic boron nitride polycrystal having a small grain size.

[0013] A cubic boron nitride polycrystal according to one embodiment of the present invention includes cubic boron nitride, the cubic boron nitride having an average grain size of not more than 150 nm, a ratio b/a being not more than 0.085 in measurement of Knoop hardness at 23° C.±5° C. under a test load of 4.9 N, the ratio b/a being a ratio between a length a of a longer diagonal line and a length b of a shorter diagonal line of a Knoop indentation.

[0014] Further, a method of producing a cubic boron nitride polycrystal according to one embodiment of the present invention includes the steps of:

[0015] preparing, as a starting material, hexagonal boron nitride powder having a grain size of not more than 0.5 μm; and

[0016] converting the hexagonal boron nitride powder into cubic boron nitride and wurtzite type boron nitride and sintering the cubic boron nitride and the wurtzite type boron nitride under temperature and pressure satisfying the following conditions:

$$P \geq 0.0000122T^2 - 0.0531T + 65.846,$$

$$T \leq 2200, \text{ and}$$

$$P \leq 25,$$

where the pressure is represented as P (GPa) and the temperature is represented as T (SC).

[0017] The foregoing and other objects, features, aspects and advantages of the present invention will become more apparent from the following detailed description of the present invention when taken in conjunction with the accompanying drawings.

BRIEF DESCRIPTION OF THE DRAWINGS

[0018] FIG. **1** illustrates a Knoop indentation.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

Description of Embodiments of the Invention of the Present Application

[0019] First, the following describes embodiments of the present invention.

[0020] As a result of diligent study to solve the above-described problem, the present inventors have found that a tough cubic boron nitride polycrystal having a fine structure can be obtained by converting hexagonal boron nitride powder having a grain size of not more than 0.5 μm into cubic boron nitride under high pressure and high temperature.

[0021] [1] Specifically, a cubic boron nitride polycrystal according to one embodiment of the present invention includes cubic boron nitride, the cubic boron nitride having an average grain size of not more than 150 nm, a ratio b/a being not more than 0.085 in measurement of Knoop hardness at 23° C.±5° C. under a test load of 4.9 N, the ratio b-a being a ratio between a length a of a longer diagonal line and a length b of a shorter diagonal line of a Knoop indentation. This cubic boron nitride polycrystal includes cubic boron nitride having a fine grain size and is a tough polycrystal.

[0022] [2] The cubic boron nitride preferably has an average grain size of not more than 100 nm. By further decreasing the average grain size in this way, the cubic boron nitride polycrystal becomes more suitable for applications requiring a small grain size.

[0023] [3] The cubic boron nitride polycrystal preferably includes not less than 0.01 volume % of wurtzite type boron nitride. Accordingly, the structure of the polycrystal will become more dense.

[0024] [4] The cubic boron nitride polycrystal preferably includes 0.01 to 0.5 volume % of compressed hexagonal boron nitride. Accordingly, crack can be prevented from being developed and toughness can be improved.

[0025] [5] For X-ray diffraction in the cubic boron nitride, a ratio $I_{(220)}/I_{(111)}$ of X-ray diffraction strength $I_{(220)}$, at a (220) plane to X-ray diffraction strength $I_{(111)}$ at a (111) plane is preferably not less than 0.1 and not more than 0.3. Accordingly, the polycrystal becomes isotropic, whereby uneven wear is reduced when applied as a tool or the like.

[0026] [6]A cutting tool according to one embodiment of the present invention includes the cubic boron nitride polycrystal described above. This cutting tool is useful to cut an iron-based material.

[0027] [7]A wear-resistant tool according to one embodiment of the present invention includes the cubic boron nitride polycrystal described above. This wear-resistant tool is useful to process an iron-based material.

[0028] [8]A grinding tool according to one embodiment of the present invention includes the cubic boron nitride polycrystal described above. This grinding tool is useful to grind an iron-based material.

[0029] [9]A method of producing a cubic boron nitride polycrystal according to one embodiment of the present invention includes the steps of:

[0030] preparing, as a starting material, hexagonal boron nitride powder having a grain size of not more than 0.5 μm; and

[0031] converting the hexagonal boron nitride powder into cubic boron nitride and wurtzite type boron nitride and sintering the cubic boron nitride and the wurtzite type boron nitride under temperature and pressure satisfying the following conditions:

$$P \geq 0.0000122T^2 - 0.0531T + 65.846,$$

$$T \leq 2200, \text{ and}$$

$$P \leq 25,$$

where the pressure is represented as P (GPa) and the temperature is represented as T (° C.). The cubic boron nitride polycrystal obtained by this production method includes cubic boron nitride having a fine grain size and is a tough polycrystal.

Details of the Embodiments of the Invention of the Present Application

[0032] The following describes an embodiment (hereinafter, referred to as "the present embodiment") of the invention of the present application more in detail.

[0033] <Cubic Boron Nitride Polycrystal>

[0034] A cubic boron nitride polycrystal according to the present embodiment includes cubic boron nitride, the cubic boron nitride having an average grain size of not more than 150 nm, a ratio b/a being not more than 0.085 in measurement of Knoop hardness at 23° C.±5° C. under a test load of 4.9 N, the ratio b/a being a ratio between a length a of a longer diagonal line and a length b of a shorter diagonal line of a Knoop indentation.

[0035] Thus, the cubic boron nitride polycrystal according to the present embodiment includes cubic boron nitride. As long as cubic boron nitride is included, an inevitable impurity may be included to such an extent that the effect of the present embodiment is exhibited. Examples of the inevitable impurity include nitrogen (N_2), hydrogen (H_2), oxygen (O_2), and the like. The polycrystal does not substantially include a binder, a sintering additive, a catalyst, and the like, which is one of advantages of the cubic boron nitride polycrystal of the present embodiment. This is because disadvantages can be overcome which result from inclusion of binder, sintering additive, and catalyst as in a conventional cubic boron nitride sintered compact.

[0036] Moreover, the cubic boron nitride polycrystal preferably contains not more than 0.1 volume % of normal pressure type boron nitride. This is because when more than 0.1 volume % of normal pressure type boron nitride is contained, strength may be decreased significantly.

[0037] It should be noted that the cubic boron nitride polycrystal is a sintered compact, but, in many cases, the term "sintered compact" usually implies that a binder is included, so that the term "polycrystal" is used in the present embodiment.

[0038] <Cubic Boron Nitride>

[0039] The cubic boron nitride included in the cubic boron nitride polycrystal of the present embodiment has a small grain size, preferably has an average particle size of not more than 150 nm, and more preferably has an average particle size of not more than 100 nm. Moreover, a smaller average particle size is more preferable and the lower limit thereof therefore does not need to be limited; however, for production, the lower limit is 20 nm.

[0040] The grain sizes in such cubic boron nitride are preferably uniform to attain no stress concentration and high strength, so that the average particle size herein preferably represents a normal distribution. When grains having large and small grain sizes are included, stress is concentrated thereon and strength accordingly becomes low, so that the average particle size preferably represents a normal distribution and is preferably uniform. It should be noted that, in the present application, the expression "grain size of cubic boron nitride" represents the grain size of a crystal grain of cubic boron nitride of the cubic boron nitride polycrystal.

[0041] The average particle size can be found by an intercept method employing a scanning electron microscope. Specifically, a scanning electron microscope (SEM) is first used to observe the cubic boron nitride polycrystal at a magnification of 1000× to 100000× so as to obtain a SEM image.

[0042] Next, a circle is drawn on the SEM image and then eight straight lines are drawn from the center of the circle to the outer circumference of the circle in a radial manner (in such a manner that intersecting angles between the straight lines become substantially equal to one another). In this case, the observation magnification and the diameter of the circle are preferably set such that the number of cubic boron nitride grains (crystal grains) on each straight line becomes about 10 to 50.

[0043] Then, the number of crystal grain boundaries of the cubic boron nitride crossed by each of the straight lines is counted, then, the length of the straight line is divided by the number thereof to find an average intercept length, then, the average intercept length is multiplied by 1.128, and the resulting value is regarded as the average grain size. It should be noted that such an average grain size is more preferably obtained in the following manner: several SEM images are used, an average grain size is found for each image through the above-described method, and the average value of the average grain sizes is suitably regarded as the average grain size. It should be also noted that in the measurement by the above-described method, the grain size of a grain (such as a crystal grain of wurtzite type boron nitride) other than the cubic boron nitride may be included; however, the measured value is regarded as the average grain size of the cubic boron nitride even though the grain size of the other grain is included.

[0044] Because the cubic boron nitride included in the cubic boron nitride polycrystal of the present embodiment has such a small grain size, it can be employed for a wide range of applications such as an application involving a large load and a micro-processing application when used for a tool or the like.

[0045] <Knoop Hardness>

[0046] In the cubic boron nitride polycrystal of the present embodiment, the ratio b/a is not more than 0.085 in measurement of Knoop hardness at 23° C.±5° C. under a test load of 4.9 N, the ratio b/a being a ratio between a length a of a longer diagonal line and a length b of a shorter diagonal line of a Knoop indentation.

[0047] The measurement of such Knoop hardness is known as one of criteria representing hardness of industrial materials as defined in, for example, JIS Z 2251, and is performed such that the Knoop indenter is pressed against a target material under a predetermined temperature and a predetermined load (test load) so as to find the hardness of the material.

[0048] Here, the Knoop indenter is an indenter made of diamond and having a shape of quadrangular prism having a bottom surface with a shape of rhomboid. The rhomboid of the bottom surface is defined to satisfy a ratio b/a of 0.141, the ratio b-a being a ratio between length a of a longer diagonal line and length b of a shorter diagonal line. Moreover, the Knoop indentation is a mark left in the target material (cubic boron nitride polycrystal in the present embodiment) at a portion on which the Knoop indenter is pressed under the above-described temperature and test load and from which the Knoop indenter is released just after that.

[0049] One feature of the cubic boron nitride polycrystal of the present embodiment lies in that the ratio b/a (not more than 0.085) in the Knoop indentation is smaller than the original ratio b/a (0.141) in the Knoop indenter. This is because the target material (i.e., cubic boron nitride polycrystal in the present embodiment) behaves elastically and experiences recovery (elastic recovery) taking place in the indentation toward elastically reverting to the original form.

[0050] Specifically, with reference to FIG. **1** conceptually showing the Knoop indentation, the above-described phenomenon is illustrated, for example, as follows: the cross section of the Knoop indenter and the Knoop indentation become the same in shape (portion indicated as "ORIGINAL KNOOP INDENTATION" in FIG. **1**) when the target material does not exhibit elastic recovery at all, but the elastic recovery is likely to take place in the direction of arrow in the FIGURE and therefore the Knoop indentation of the present embodiment is in the form of a rhomboid indicated by a solid line in the FIGURE. In other words, it is indicated that the value of the ratio b/a becomes smaller as the reversion in the direction of arrow in the FIGURE becomes larger, and as this value is smaller, the elastic recovery (elastic property) is larger.

[0051] Apparently, because the cubic boron nitride polycrystal of the present embodiment has the above-described ratio b/a in the Knoop indentation, the cubic boron nitride polycrystal has a large elastic recovery property and has high toughness due to the high elastic recovery, so that the cubic boron nitride polycrystal is a tough cubic boron nitride polycrystal. As described above, the ratio b/a in the Knoop indentation in the present embodiment serves as an index indicating how large or small the elastic recovery of the cubic boron nitride polycrystal is.

[0052] In addition, it can be said that a smaller ratio b/a in Knoop indentation is preferable because the elastic recovery becomes larger as the ratio b/a becomes smaller, so that the lower limit of the ratio b/a does not particularly need to be limited; however, the elastic property becomes large when the elastic recovery becomes too large, in other words, elastic deformation becomes large during processing to result in deteriorated workability when used as a tool. In view of this, the lower limit value is preferably set at 0.060. The ratio b/a in such a Knoop indentation is more preferably 0.070 to 0.080.

[0053] <Wurtzite Type Boron Nitride>

[0054] The cubic boron nitride polycrystal of the present embodiment may include wurtzite type boron nitride (wBN), and preferably includes not less than 0.01 volume % of such wurtzite type boron nitride. Accordingly, the structure of the polycrystal become more dense. It should be noted that in view of characteristics of the wurtzite type boron nitride, the upper limit of the content of the wurtzite type boron nitride is not more than 65 volume %. Existence of the wurtzite type boron nitride provides an effect of hindering development of cracks and improving toughness, but the wurtzite type boron nitride is a metastable phase during transition from hexagonal boron nitride to cubic boron nitride and therefore has characteristics such that stability is inferior to that of cubic boron nitride and wear resistance is low. A more preferable range of the content of the wurtzite type boron nitride is 0.01 to 20 volume %.

[0055] When the cubic boron nitride polycrystal includes the wurtzite type boron nitride, the cubic boron nitride polycrystal is configured such that a plurality of crystal grains of the cubic boron nitride and a plurality of crystal grains of the wurtzite type boron nitride are distributed to each other. Moreover, the crystal grains of the cubic boron nitride are bonded to one another firmly, the crystal grains of the wurtzite type boron nitride are bonded to one another firmly, and the crystal grains of the cubic boron nitride and wurtzite type boron nitride are bonded to one another firmly, thereby attaining a dense structure.

[0056] It should be noted that in the cubic boron nitride polycrystal containing not less than 0.01 volume % of the wurtzite type boron nitride with the remainder being composed of the cubic boron nitride and an inevitable impurity, the cubic boron nitride has an average grain size of about 28 nm to 85 nm and the ratio b/a in the Knoop indentation is not more than 0.084.

[0057] <Compressed Hexagonal Boron Nitride>

[0058] The cubic boron nitride polycrystal of the present embodiment may include compressed hexagonal boron nitride, and preferably includes 0.01 to 0.5 volume % of such compressed hexagonal boron nitride. This can provide a function of hindering development of cracks and improving toughness. Moreover, because the existence of the compressed hBN is permitted, sintering can be achieved in a wide temperature range, thus leading to improved productivity. However, if more than 0.5 volume % of the compressed hBN is included, stress concentration in the compressed hBN may become large to decrease the strength. Therefore, when the cubic boron nitride polycrystal further includes the compressed hBN, the upper limit thereof is 0.5 volume %. The volume content of the compressed hBN is more preferably not less than 0.01 volume % and not more than 0.1 volume %, and is particularly preferably not less than 0.05 volume % and not more than 0.1 volume %.

[0059] <X-Ray Diffraction>

[0060] For X-ray diffraction in the cubic boron nitride included in the cubic boron nitride polycrystal of the present embodiment, a ratio $I_{(220)}/I_{(111)}$ of X-ray diffraction strength $I_{(220)}$ at a (220) plane to X-ray diffraction strength $I_{(111)}$ at a (111) plane is preferably not less than 0.1 and not more than 0.3. Accordingly, the polycrystal becomes isotropic, whereby uneven wear is reduced when applied to a tool or the like.

[0061] When the ratio $I_{(220)}/I_{(111)}$ falls out of the above-described range, orientation is achieved in the polycrystal, resulting in anisotropy in the polycrystal. In this case, there is distribution in strength in the polycrystal, thus providing a surface having high strength and a surface having weak strength. Therefore, it is inappropriate to use the polycrystal for a tool. Particularly, in the case of a rotation tool such as an end mill, the surfaces thereof are divided into a surface that is readily worn or is readily chipped and a surface that is unlikely worn or is unlikely chipped, thus resulting in uneven wear. In order to overcome such a shortcoming, it is preferable to achieve isotropy in the polycrystal.

[0062] The ratio $I_{(220)}/I_{(111)}$ is more preferably not less than 0.15 and not more than 0.25.

[0063] <Application>

[0064] Because the cubic boron nitride polycrystal of the present embodiment includes cubic boron nitride (crystal grain) having a fine grain size and is tough, it is suitable to use the cubic boron nitride polycrystal for a cutting tool, a wear-resistant tool, a grinding tool, or the like. In other words, each of the cutting tool, wear-resistant tool, and grinding tool of the present embodiment is provided with the above-described cubic boron nitride polycrystal.

[0065] It should be noted that each of the tools may be entirely constituted of the cubic boron nitride polycrystal, or only a portion thereof (for example, an edge portion in the case of the cutting tool) may be constituted of the cubic boron nitride polycrystal. Moreover, a coating film may be formed on a surface of each of the tools.

[0066] Here, examples of the cutting tool includes a drill, an end mill, an edge-replaceable cutting insert for drill, an edge-replaceable cutting insert for end mill, an edge-replaceable cutting insert for milling, an edge-replaceable cutting insert for turning, a metal saw, a gear cutting tool, a reamer, a tap, a cutting bite, and the like.

[0067] Further, examples of the wear-resistant tool include a die, a scriber, a scribing wheel, a dresser, and the like.

[0068] Further, examples of the grinding tool include a grinding stone and the like.

[0069] <Production Method>

[0070] The method of producing the cubic boron nitride polycrystal according to the present embodiment includes the steps of:

[0071] preparing, as a starting material, hexagonal boron nitride powder having a grain size of not more than 0.5 μm (hereinafter, also referred to as "preparing step"); and

[0072] converting the hexagonal boron nitride powder into cubic boron nitride and wurtzite type boron nitride and sintering the cubic boron nitride and the wurtzite type boron nitride under temperature and pressure satisfying the following conditions (hereinafter, also referred to as "sintering step"):

$$P \geq 0.0000122T^2 - 0.0531T + 65.846,$$

$$T \leq 2200, \text{ and}$$

$$P \leq 25,$$

where the pressure is represented as P (GPa) and the temperature is represented as T (° C.).

[0073] With the production method, the cubic boron nitride polycrystal described above can be produced. Specifically, the cubic boron nitride polycrystal obtained by this production method includes cubic boron nitride having a fine grain size (i.e., an average particle size of not more than 150 nm) and is a tough polycrystal.

[0074] In the preparing step, the grain size of hexagonal boron nitride powder, which is employed as a starting material, may have a slightly larger grain size than the average grain size of the cubic boron nitride of the cubic boron nitride polycrystal to be obtained. This is because during transition from the hexagonal boron nitride to the cubic boron nitride, binding between atoms in the hBN is disconnected and the atoms are rearranged and recombined, with the result that the grain size of the cubic boron nitride becomes smaller than the grain size of the raw material. However, when the grain size of the raw material is small, there occur a large number of grain boundaries having no intrinsic binding between the atoms in the hBN, with the result that the grain size of the cubic boron nitride after the conversion becomes small. Conversely, when the grain size of the raw material is large, the grain size of the cubic boron nitride becomes large, resulting in a large value of the ratio b/a. Therefore, the grain size of the hexagonal boron nitride powder is set at not more than 0.5 μm and the lower limit value thereof is 0.05 μm due to the production reason. A more preferable grain size is not less than 0.1 μm and not more than 0.5 μm.

[0075] It should be noted that the grain size of the hexagonal boron nitride powder refers to an average grain size measured by a laser diffraction scattering method employing laser light.

[0076] As long as the above-described hexagonal boron nitride powder is prepared, such a preparing step is not limited in terms of its manner, and can be performed, for example, in such a manner that hexagonal boron nitride powder can be produced by a conventionally known synthesis method or in such a manner that commercially available hexagonal boron nitride powder can be acquired.

[0077] Moreover, because problems such as grain growth taking place when the temperature is high and unconverted hBN remaining when the temperature is low lead to the ratio b/a exceeding 0.085, pressure P (GPa) and temperature T (° C.) in the above-described sintering step are required to satisfy the following conditions.

$$P \geq 0.0000122T^2 - 0.0531T + 65.846,$$

$$T \leq 2200, \text{ and}$$

$$P \leq 25,$$

[0078] Here, temperature T (° C.) is not particularly limited as long as the cubic boron nitride polycrystal can be obtained at the temperature, and the lower limit value thereof does not need to be defined. More preferably, this temperature T (° C.) is 1300 to 2100° C.

[0079] Likewise, pressure P (GPa) is not particularly limited as long as the cubic boron nitride polycrystal can be obtained at the pressure, and the lower limit value thereof does not need to be defined. More preferably, this pressure P (GPa) is 10 to 20 GPa.

[0080] When the sintering step employing the temperature and pressure in the respective suitable ranges and satisfying the above-described relational expressions is performed, the ratio b/a in the Knoop indentation of the obtained cubic boron nitride polycrystal is not more than 0.085, preferably, 0.072 to 0.084.

[0081] It should be noted that the above-described temperature and pressure in the sintering step are applied for 5 to 20 minutes. If it is shorter than 5 minutes, the sintering is insufficient, whereas if it is longer than 20 minutes, there is no difference in sintering state, which is disadvantageous economically. More preferably, they are applied for 10 to 20 minutes.

[0082] While such a sintering step is a step of converting the hexagonal boron nitride powder into the cubic boron nitride and the wurtzite type boron nitride and sintering the cubic boron nitride and the wurtzite type boron nitride, the hexagonal boron nitride powder is solely and directly converted into the cubic boron nitride and wurtzite type boron nitride without using a sintering additive and a catalyst and this conversion is normally performed at the same time as the sintering.

[0083] The cubic boron nitride polycrystal obtained by the production method includes the cubic boron nitride having a fine grain size, exhibits an elastic behavior, and is therefore a tough polycrystal improved in chipping resistance. Hence, the cubic boron nitride polycrystal can be used suitably for a cutting tool, a wear-resistant tool, a grinding tool, and the like, which are employed for high-speed micro-processing involving a large load.

EXAMPLES

[0084] While the present invention will be described in more detail with reference to Examples, the present invention is not limited thereto.

Examples 1 to 5

[0085] Each of cubic boron nitride polycrystals according to Examples 1 to 5 was produced by the following method. First, as a starting material, hexagonal boron nitride powder having a grain size of 0.5 μm (indicated as "Fine-Grain hBN Powder" in the column "Starting Material" in Table 1 below) was prepared (preparing step).

[0086] Next, the hexagonal boron nitride powder prepared as above was introduced into a capsule made of metal having a high melting point, and was held for 20 minutes in temperature and pressure described in Table 1 (column "Synthesis Condition") using a ultra-high pressure/high-temperature generating device, thereby converting the hexagonal boron nitride powder into cubic boron nitride and wurtzite type boron nitride and sintering them (sintering step). Accordingly, the cubic boron nitride polycrystal was obtained.

[0087] It should be noted that the temperature and pressure in Table 1 satisfy the following condition. $P \geq 0000122T^2 - 0$ $0531T + 65.846$.

Comparative Example 1

[0088] A cubic boron nitride polycrystal according to Comparative Example 1 was produced by the following method. First, as a starting material, hexagonal boron nitride powder having a grain size of 0.5 μm (indicated as "Fine-Grain hBN Powder" in the column "Starting Material" in Table 1 below) was prepared (preparing step).

[0089] Next, the hexagonal boron nitride powder prepared as above was introduced into a capsule made of metal having a high melting point, and was held for 20 minutes in temperature and pressure described in Table 1 (column "Synthesis Condition") using a ultra-high pressure/high-temperature generating device, thereby converting the hexagonal boron nitride powder into cubic boron nitride and sintering it (sintering step). Accordingly, the cubic boron nitride polycrystal was obtained.

[0090] Regarding the above-described conditions, the temperature is 2400° C., which does not satisfy the condition of $T \leq 2200$.

Comparative Example 2

[0091] A cubic boron nitride polycrystal according to Comparative Example 2 was produced by the following method. First, as a starting material, hexagonal boron nitride powder having a grain size of 0.5 μm (indicated as "Fine-Grain hBN Powder" in the column "Starting Material" in Table 1 below) was prepared (preparing step).

[0092] Next, the hexagonal boron nitride powder prepared as above was introduced into a capsule made of metal having a high melting point, and was held for 20 minutes in temperature and pressure described in Table 1 (column "Synthesis Condition") using an upward/downward pressure-application belt-type high pressure/high-temperature generating device, thereby converting the hexagonal boron nitride powder into cubic boron nitride and wurtzite type boron nitride and sintering them (sintering step). Accordingly, the cubic boron nitride polycrystal was obtained.

[0093] It should be noted that the above-described condition does not satisfy the following relation: $P \geq 0.0000122T^2 - 0.0531T + 65.846$.

Comparative Example 3

[0094] A cubic boron nitride polycrystal according to Comparative Example 3 was produced by the following method. First, as a starting material, hexagonal boron nitride powder having a grain size of not more than 5 μm (indicated as "Coarse-Grain hBN Powder" in the column "Starting Material" in Table 1 below) was prepared (preparing step).

[0095] Next, the hexagonal boron nitride powder prepared as above was introduced into a capsule made of metal having a high melting point, and was held for 20 minutes in temperature and pressure described in Table 1 (column "Synthesis Condition") using a ultra-high pressure/high-temperature generating device, thereby converting the hexagonal boron nitride powder into cubic boron nitride and wurtzite type boron nitride and sintering them (sintering step). Accordingly, the cubic boron nitride polycrystal was obtained.

[0096] It should be noted that the conditions above are different from those of the Examples in that the grain size of the starting material is not more than 5 μm, which is larger than that of the starting material of each of the Examples.

Comparative Example 4

[0097] A cubic boron nitride sintered compact according to Comparative Example 4 was produced by the following method. First, as a starting material, cubic boron nitride powder having an average grain size of not more than 2 μm and Co-based metal binder powder (indicated as "cBN Powder/Metal Binder Powder" in the column "Starting Material" in Table 1 below) were prepared (preparing step).

[0098] Next, the cubic boron nitride powder and Co-based metal binder powder prepared as above were introduced into a capsule made of metal having a high melting point, and were sintered by holding them for 20 minutes using an ultra-high pressure/high-temperature generating device under temperature and pressure described in Table 1 (column "Synthesis Condition") (sintering step). Accordingly, the cubic boron nitride sintered compact was obtained.

[0099] Regarding the conditions above, the starting material was different from that of each of the Examples.

[0100] <Evaluation>

[0101] A technique described below was employed to measure composition, X-ray diffraction, grain size, and ratio b/a in the Knoop indentation in each of the cubic boron nitride polycrystals of Examples 1 to 5 and Comparative Examples 1 to 3 and the cubic boron nitride sintered compact of Comparative Example 4, which had been obtained in the manners mentioned above.

[0102] <Composition>

[0103] The cubic boron nitride (cBN), compressed hexagonal boron nitride (hBN), and wurtzite type boron nitride (wBN) included in each of the cubic boron nitride polycrystals and the cubic boron nitride sintered compact were identified using an X-ray diffraction device. This device had an X-ray radiation source, which was Cu and provided Kα ray having a wavelength of 1.54 Å. The result is shown in the column "Composition" of Table 1.

[0104] <X-Ray Diffraction>

[0105] For cubic boron nitride included in each of the cubic boron nitride polycrystals and the cubic boron nitride sintered compact, the X-ray diffraction device was employed to find a ratio $I_{(220)}/I_{(111)}$ of X-ray diffraction strength $I_{(220)}$ at a (220) plane to an X-ray diffraction strength $I_{(111)}$ at a (111) plane. This device had an X-ray radiation source, which was Cu and provided Kα ray having a wavelength of 1.54 Å. The result is shown in the column "XRD $I_{(220)}/I_{(111)}$" of Table 1.

[0106] <Grain Size>

[0107] An intercept method employing a scanning electron microscope was used to find an average grain size of cubic boron nitride included in each of the cubic boron nitride polycrystals and the cubic boron nitride sintered compact.

[0108] Specifically, a scanning electron microscope (SEM) was first used to observe each of the cubic boron nitride polycrystals and the cubic boron nitride sintered compact so as to obtain a SEM image.

[0109] Next, a circle was drawn on the SEM image and then eight straight lines are drawn from the center of the circle to the outer circumference of the circle in a radial manner (in such a manner that intersecting angles between the straight lines become substantially equal to one another). In this case, the observation magnification and the diameter of the circle were set such that the number of cubic boron nitride grains on each straight line became about 10 to 50.

[0110] Then, the number of crystal grain boundaries of the cubic boron nitride crossed by each of the straight lines was counted, then, the length of the straight line was divided by the number thereof to find an average intercept length, then, the average intercept length is multiplied by 1.128, and the resulting value was regarded as the average grain size.

[0111] It should be noted that the magnification of the SEM image was 30000×. This is because with a magnification equal to or less than this magnification, the number of grains in the circle is increased, it becomes difficult to see grain boundaries, and the number of grain boundaries is wrongly counted, and a plate structure is highly likely to be included when drawing the lines. This is also because with a magnification equal to or more than this, the number of grains in the circle is too small to accurately calculate the average particle size. For each of Comparative Examples 1 and 4, the grain size was too large, so that a magnification of 3000× was employed.

[0112] Moreover, three SEM images captured in one sample at separate portions were used for each of the Examples and Comparative Examples, an average grain size was found by the above-described method for each SEM image, and the average value of the three average grain sizes obtained was regarded as an average grain size. The result is shown in the column “Average Grain Size” in Table 1.

[0113] <Ratio b/a in Knoop Indentation and Hardness>

[0114] For each of the cubic boron nitride polycrystals and the cubic boron nitride sintered compact, in order to measure the ratio b/a in the Knoop indentation, Knoop hardness was measured under the following conditions.

[0115] That is, a micro Knoop indenter was used as a Knoop indenter, and the Knoop hardness was measured five times at 23° C.±5° C. under a test load of 4.9 N. Then, for each measurement, a laser microscope was used to measure the ratio b/a between length a of a longer diagonal line and length b of a shorter diagonal line of the Knoop indentation, and the average value thereof was regarded as the ratio b/a in the Knoop indentation. The result is shown in the column “Ratio b/a” in Table 1.

[0116] Moreover, as the hardness of each sample, the average value of three values except the smallest and largest values in the results of measurements performed five times as described above was found and the result is shown in the column “Hardness” in Table 1.

TABLE 1

	Starting Material	Synthesis Condition		Composition (Volume %)			XRD	Average Grain Size (nm)	Ratio b/a	Hardness (GPa)
		Pressure (GPa)	Temperature (° C.)	cBN	hBN	wBN	$I_{(220)}/I_{(111)}$			
Example 1	Fine-Grain hBN Powder	20	1300	39.5	0	60.5	0.18	28	0.072	43
Example 2	Fine-Grain hBN Powder	20	1500	87.5	0	12.5	0.29	48	0.075	45
Example 3	Fine-Grain hBN Powder	15	1700	92.1	0	7.9	0.10	66	0.078	46
Example 4	Fine-Grain hBN Powder	10	1800	97.83	0.07	2.1	0.12	79	0.081	45
Example 5	Fine-Grain hBN Powder	10	2100	99.0	0	1.0	0.23	85	0.084	44
Comparative Example 1	Fine-Grain hBN Powder	10	2400	100	0	0	0.20	1030	0.102	43
Comparative Example 2	Fine-Grain hBN Powder	7.7	2300	99.91	0.09	0	0.03	453	0.096	44
Comparative Example 3	Coarse-Grain hBN Powder	10	2100	98.5	0	1.5	0.21	191	0.091	43
Comparative Example 4	cBN Powder/Metal Binder Powder	7	1800	100 (Excluding Binder)	0	0	—	2000	0.113	35

[0117] As shown in Table 1, it was confirmed that each of Examples 1 to 5 contains 1.0 to 60.5 volume % of wurtzite type boron nitride (wBN). Moreover, the average grain size of cubic boron nitride of each of Examples 1 to 5 was 28 to 85 nm. On this occasion, the ratio b/a in the Knoop indentation in each of Examples 1 to 5 was 0.072 to 0.084 On the other hand, the average grain size of the cubic boron nitride of Comparative Example 1 was 1030 nm, which was larger than that of each of Examples 1 to 5. Meanwhile, the ratio b/a in the Knoop indentation of Comparative Example 1 was 0.102, which apparently indicated that an amount of elastic recovery was smaller than that in each of Examples 1 to 5 and therefore the elastic property was also small.

[0118] Moreover, in Comparative Example 2, the synthesis condition did not satisfy the relation $P \geq 0.0000122T^2 - 0.0531T + 65.846$, and the ratio $I_{(220)}/I_{(111)}$ was 0.03 in the X-ray diffraction for cubic boron nitride, so that orientation is high to result in non-isotropy.

[0119] Moreover, Comparative Example 3, which employed the hexagonal boron nitride powder having a grain size of 5 μm as the raw material, had an average grain size of 191 nm, which was larger than that of each of Examples 1 to 5. On this occasion, the ratio b/a in the Knoop indentation was 0.091, which apparently indicated that an amount of elastic recovery is smaller than that in each of Examples 1 to 5 and therefore the elastic property is also small.

[0120] Moreover, in Comparative Example 4, which employed the cubic boron nitride powder and the binder as the raw material, the ratio b/a in the Knoop indentation was 0.113, which apparently indicated that an amount of elastic recovery is smaller than that of each of Examples 1 to 5 and therefore the elastic property was also small.

[0121] Further, each of the cubic boron nitride polycrystals and cubic boron nitride sintered compact of each of the Examples and the Comparative Examples was attached to the tip of a ball end mill tool having a tip diameter of 0.5 mm, and then cutting performance thereof was evaluated. A hardened steel of HRC60 was prepared as a material to be cut, and was cut by 24 m under conditions that a rotating speed was 60000 rpm, a cutting speed was 200 mm/min, a cut-in amount was 5 μm, and a feed amount was 3 pun. A wear amount of the tool upon completion of the cutting is shown as “Tool Wear Relative Ratio” in Table 2.

TABLE 2

	Tool Wear Relative Ratio
Example 1	1.8
Example 2	1
Example 3	1.2
Example 4	2.2
Example 5	2
Comparative Example 1	Cutting Edge Greatly Chipped; Processing Discontinued
Comparative Example 2	Cutting Edge Greatly Chipped; Processing Discontinued
Comparative Example 3	3.8

TABLE 2-continued

	Tool Wear Relative Ratio
Comparative Example 4	7.8

[0122] Assuming that Example 2 providing the smallest wear amount was a reference, the wear amounts of the Examples were 1 to 2.2 as shown in Table 2. On the other hand, in Comparative Examples 1 and 2, great chippings took place at cutting lengths of 12 m and 16 m respectively and therefore the processing was discontinued. Moreover, Comparative Examples 3 and 4 were worn greatly, specifically, their respective wear amounts were 3.8 times and 7.8 times as large as that of Example 2. Thus, it was confirmed that the cubic boron nitride polycrystal of each of the Examples was tougher than the cubic boron nitride polycrystals and cubic boron nitride sintered compact of the Comparative Examples.

[0123] Heretofore, the embodiments and examples of the present invention have been illustrated, but it has been initially expected to appropriately combine the configurations of the embodiments and examples and modify them in various manners.

[0124] Although the present invention has been described and illustrated in detail, it is clearly understood that the same is by way of illustration and example only and is not to be taken by way of limitation, the scope of the present invention being interpreted by the terms of the appended claims.

What is claimed is:

1. A cubic boron nitride polycrystal comprising cubic boron nitride,

said cubic boron nitride having an average grain size of not more than 150 nm,

a ratio b/a being not more than 0.085 in measurement of Knoop hardness at 23° C.±5° C. under a test load of 4.9 N, said ratio b/a being a ratio between a length a of a longer diagonal line and a length b of a shorter diagonal line of a Knoop indentation.

2. The cubic boron nitride polycrystal according to claim **1**, wherein said cubic boron nitride has an average grain size of not more than 100 nm.

3. The cubic boron nitride polycrystal according to claim **1**, wherein said cubic boron nitride polycrystal includes not less than 0.01 volume % of wurtzite type boron nitride.

4. The cubic boron nitride polycrystal according to claim **1**, wherein said cubic boron nitride polycrystal includes 0.01 to 0.5 volume % of compressed hexagonal boron nitride.

5. The cubic boron nitride polycrystal according to claim **1**, wherein for X-ray diffraction in said cubic boron nitride, a ratio $I_{(220)}/I_{(111)}$ of X-ray diffraction strength $I_{(220)}$ at a (220) plane to X-ray diffraction strength $I_{(111)}$ at a (111) plane is not less than 0.1 and not more than 0.3.

6. A cutting tool comprising the cubic boron nitride polycrystal recited in claim **1**.

7. A wear-resistant tool comprising the cubic boron nitride polycrystal recited in claim **1**.

8. A grinding tool comprising the cubic boron nitride polycrystal recited in claim **1**.

9. A method of producing a cubic boron nitride polycrystal, comprising the steps of:

preparing, as a starting material, hexagonal boron nitride powder having a grain size of not more than 0.5 μm; and

converting said hexagonal boron nitride powder into cubic boron nitride and wurtzite type boron nitride and sintering said cubic boron nitride and said wurtzite type boron nitride under temperature and pressure satisfying the following conditions:

$$P \geq 0.0000122T^2 - 0.0531T + 65.846,$$

$$T \leq 2200, \text{ and}$$

$$P \leq 25,$$

where the pressure is represented as P (GPa) and the temperature is represented as T (° C.).

* * * * *