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(71) Applicant: **DIC CORPORATION** [JP/JP]; 35-58, Sakashita 3-chome, Itabashi-ku, Tokyo 174-8520 (JP).

(72) Inventor; and

(71) Applicant (for SC only): **YANG, Shaowei** [CN/CN]; c/o Qingdao DIC Finechemicals Co., Ltd., A Area, Hit Industrial City, 177 Zhuzhou Road, Qingdao, Shandong 266101 (CN).

(72) Inventors: **YUAN, Jianjun**; c/o DIC Corporation, Central Research Laboratories, 631, Sakado, Sakura-shi, Chiba 285-8668 (JP). **TABUCHI, Minoru**; c/o DIC Corporation, Central Research Laboratories, 631, Sakado, Sakura-shi, Chiba 285-8668 (JP). **YAGI, Naoto**; c/o DIC Corporation, Central Research Laboratories, 631, Sakado, Sakura-shi, Chiba 285-8668 (JP). **SUN, Xiao**; c/o Qingdao DIC Finechemicals Co., Ltd., A Area, Hit Industrial City, 177 Zhuzhou Road, Qingdao, Shandong 266101 (CN). **FENG, Yuanquan**; c/o Qingdao DIC Finechemicals Co., Ltd., A Area, Hit Industrial City, 177 Zhuzhou Road, Qingdao, Shandong 266101 (CN). **ZHAO, Wei**; c/o Qingdao DIC Finechemicals Co., Ltd., A Area, Hit Industrial City, 177 Zhuzhou Road, Qingdao, Shandong 266101 (CN). **GUO, Jian**; c/o Qingdao DIC Finechemicals Co., Ltd., A Area,

Hit Industrial City, 177 Zhuzhou Road, Qingdao, Shandong 266101 (CN).

(74) Agent: **LINDA LIU & PARTNERS**; F16 Tower C, Beijing Global Trade Center, 36 North Third Ring East Road, Dongcheng District, Beijing 100013 (CN).

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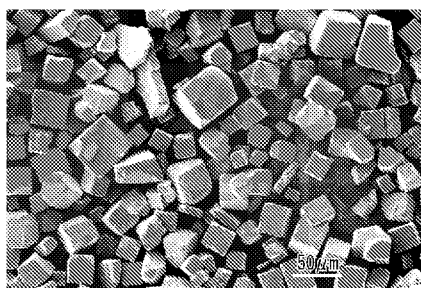
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(54) Title: TANTALATE PARTICLES AND METHOD FOR PRODUCING TANTALATE PARTICLES

FIG. 1

EXAMPLE 1



(57) Abstract: Tantalate particles containing molybdenum and represented by $K_xNa_{(1-x)}Ta_yO_z$ are disclosed, where $x = 0$ to 1 , $y = 1$ to 10 , and $z = 3$ to 20 . A method for producing the tantalate particles, and the method includes firing a tantalum compound in the presence of a molybdenum compound and at least one of a potassium compound and a sodium compound are also disclosed.



[DESCRIPTION]

[Title of Invention]

TANTALATE PARTICLES AND METHOD FOR PRODUCING TANTALATE PARTICLES

[Technical Field]

[0001]

The present invention relates to tantalate particles and a method for producing tantalate particles.

[Background Art]

[0002]

Alkali metal tantalate salts are widely used as piezoelectric materials, fillers, catalysts, semiconductor photoelectrodes, and the like.

[0003]

PTL 1 discloses a method for producing tantalate crystal particles that have a layered perovskite type structure and are represented by a specific formula. The method for producing tantalate crystal particles includes mixing raw materials with flux and heating the mixture, thereby precipitating and growing a crystal. Furthermore, PTL 1 discloses an example in which the flux contains potassium chloride or strontium chloride.

[0004]

PTL 2 discloses a catalyst composition that is an application example for use in photocatalytic reduction of carbon dioxide. The catalyst composition includes sodium tantalate (NaTaO_3), which is a base catalyst; a modifying agent; and at least one co-catalyst.

[Citation List]

[Patent Literature]

[0005]

[PTL 1]

Japanese Unexamined Patent Application Publication No. 2009-190927

[PTL 2]

Japanese Unexamined Patent Application Publication

(Translation of PCT Application) No. 2016-524534

[Summary of Invention]

[Technical Problem]

[0006]

However, regarding the tantalate particles and the production methods therefor of the related art, the discoveries made are limited, and, therefore, there is still room for investigation.

[0007]

The present invention has been made to solve the problem, and an object of the present invention is to provide tantalate particles having a controlled particle shape and excellent properties.

[Solution to Problem]

[0008]

The present inventors diligently performed studies to achieve the object and, consequently, found that by using a molybdenum compound as flux, a crystal shape of the tantalate particles that are to be produced can be readily controlled, and molybdenum-containing tantalate particles can be produced. Accordingly, the present inventors completed the present invention. Specifically, the present invention encompasses the following aspects.

[0009]

(1) Tantalate particles containing molybdenum and represented by $K_xNa_{(1-x)}Ta_yO_z$, where $x = 0$ to 1 , $y = 1$ to 10 , and $z = 3$ to 20 .

(2) The tantalate particles according to (1), wherein the tantalate particles include at least one type of particles selected from the group consisting of $K_xNa_{(1-x)}TaO_3$ particles, where $x = 0$ to 1 ; $Na_2Ta_4O_{11}$ particles; and $K_2Ta_4O_{11}$ particles.

(3) The tantalate particles according to (2), wherein the tantalate particles include at least one type of particles selected from the group consisting of $K_xNa_{(1-x)}TaO_3$ particles having a cubic shape, where $x = 0$ to 1 ; $Na_2Ta_4O_{11}$ particles

having a polyhedral shape; and $K_2Ta_4O_{11}$ particles having a polyhedral shape.

(4) The tantalate particles according to (2) or (3), wherein the tantalate particles include the $K_xNa_{(1-x)}TaO_3$ particles, the $K_xNa_{(1-x)}TaO_3$ particles have a cubic shape, and the $K_xNa_{(1-x)}TaO_3$ particles have a particle size of 0.1 to 100 μm .

(5) The tantalate particles according to (2) or (3), wherein the tantalate particles include the $Na_2Ta_4O_{11}$ particles, the $Na_2Ta_4O_{11}$ particles have a polyhedral shape, and the $Na_2Ta_4O_{11}$ particles have a particle size of 1 to 1000 μm .

(6) The tantalate particles according to (2) or (3), wherein the tantalate particles include the $K_2Ta_4O_{11}$ particles, the $K_2Ta_4O_{11}$ particles have a polyhedral shape, and the $K_2Ta_4O_{11}$ particles have a particle size of 1 to 1000 μm .

(7) The tantalate particles according to any one of (1) to (6), wherein a content of the molybdenum in the tantalate particles, as determined by XRF analysis of the tantalate particles, is 0.01 to 20 mass% in terms of a content percentage calculated as MoO_3 , relative to a total mass of the tantalate particles taken as 100 mass%.

(8) The tantalate particles according to any one of (1) to (7), wherein a content of tantalum in the tantalate particles, as determined by XRF analysis of the tantalate particles, is 50 to 99 mass% in terms of a content percentage calculated as Ta_2O_5 , relative to a total mass of the tantalate particles taken as 100 mass%.

(9) The tantalate particles according to any one of (1) to (8), wherein a content of at least one of potassium and sodium in the tantalate particles, as determined by XRF analysis of the tantalate particles, is 0.5 to 40 mass% in terms of a total content percentage calculated as at least one of K_2O and Na_2O , relative to a total mass of the tantalate particles taken as 100 mass%.

(10) A method for producing tantalate particles, the tantalate particles being the tantalate particles according to any one of (1) to (9), the method including firing a tantalum compound in the presence of a molybdenum compound and at least one of a potassium compound and a sodium compound.

(11) The method for producing tantalate particles according to (10), wherein the molybdenum compound is at least one compound selected from the group consisting of molybdenum trioxide, potassium molybdate, and sodium molybdate.

(12) The method for producing tantalate particles according to (10) or (11), wherein the sodium compound is sodium carbonate or sodium molybdate, and the potassium compound is potassium carbonate or potassium molybdate.

(13) The method for producing tantalate particles according to any one of (10) to (12), wherein a firing temperature for firing the tantalum compound is 700 to 1500°C.

(14) The method for producing tantalate particles according to any one of (10) to (13), the method including the steps of forming a mixture by mixing together the tantalum compound, the molybdenum compound, and the at least one of the potassium compound and the sodium compound; and firing the mixture, wherein a molar ratio of molybdenum atoms to tantalum atoms in the mixture is 0.01 to 5.

[Advantageous Effects of Invention]

[0010]

The present invention provides tantalate particles having excellent properties attributable to molybdenum and having a controlled particle shape.

[Brief Description of Drawings]

[0011]

[Fig. 1]

Fig. 1 is an SEM image of tantalate particles of Example 1.

[Fig. 2]

Fig. 2 is an SEM image of tantalate particles of Example 2.

[Fig. 3]

Fig. 3 is an SEM image of tantalate particles of Example 3.

[Fig. 4]

Fig. 4 is an SEM image of tantalate particles of Example 4.

[Fig. 5]

Fig. 5 is an SEM image of tantalate particles of Example 5.

[Fig. 6]

Fig. 6 is an SEM image of tantalate particles of Example 6.

[Fig. 7]

Fig. 7 is an SEM image of tantalum oxide particles of Comparative Example 1.

[Fig. 8]

Fig. 8 shows X-ray diffraction (XRD) patterns of the tantalate particles of Examples 1 to 3.

[Fig. 9]

Fig. 9 shows X-ray diffraction (XRD) patterns of the tantalate particles of Examples 4 to 6.

[Fig. 10]

Fig. 10 is an SEM image of tantalate particles of Example 7.

[Fig. 11]

Fig. 11 is an SEM image of tantalate particles of Example 8.

[Fig. 12]

Fig. 12 shows X-ray diffraction (XRD) patterns of the tantalate particles of Examples 7 and 8.

[Fig. 13]

Fig. 13 is an SEM image of particles of Example 9.

[Fig. 14]

Fig. 14 is an SEM image of particles of Example 10.

[Fig. 15]

Fig. 15 is an SEM image of particles of Example 11.

[Fig. 16]

Fig. 16 shows an X-ray diffraction (XRD) pattern of the particles of Example 9.

[Fig. 17]

Fig. 17 shows X-ray diffraction (XRD) patterns of the

particles of Examples 10 to 11.

[Description of Embodiments]

[0012]

An embodiment of tantalate particles of the present invention and an embodiment of a method of the present invention for producing tantalate particles will be described below.

[0013]

[Tantalate Particles]

According to an embodiment, tantalate particles are tantalate particles containing molybdenum and represented by $K_xNa_{(1-x)}Ta_yO_z$, where $x = 0$ to 1 , $y = 1$ to 10 , and $z = 3$ to 20 .

[0014]

The tantalate particles of the embodiment contain a tantalate compound represented by $K_xNa_{(1-x)}Ta_yO_z$. In the formula of $K_xNa_{(1-x)}Ta_yO_z$, $0 \leq x \leq 1$ is satisfied. When $0 < x < 1$, $K_xNa_{(1-x)}Ta_yO_z$ is potassium sodium tantalate. When $x = 0$, $K_xNa_{(1-x)}Ta_yO_z$ is sodium tantalate ($NaTa_yO_z$). When $x = 1$, $K_xNa_{(1-x)}Ta_yO_z$ is potassium tantalate (KTa_yO_z).

[0015]

The tantalate particles of the embodiment contain molybdenum and have excellent properties attributable to molybdenum, such as excellent catalytic activity. In the tantalate particles of the embodiment, the molybdenum present is derived from a molybdenum compound used in a production method, which will be described later.

Furthermore, excellent control of a particle shape of the tantalate particles of the embodiment can be achieved by the use of a molybdenum compound in the production method described later.

[0016]

Regarding the molybdenum present in the tantalate particles of the embodiment, a state of existence and an amount of the molybdenum are not particularly limited. Examples of molybdenum that may be present in the tantalate particles

include molybdenum metal, molybdenum oxide, and partially reduced molybdenum compounds. It can be assumed that the molybdenum present in the tantalate particles is in the form of MoO_3 . In addition to or in place of MoO_3 , one or more other forms of molybdenum, such as MoO_2 and MoO , may be present in the tantalate particles.

[0017]

The form in which the molybdenum is present is not particularly limited, and any of the following forms are possible: a form in which molybdenum adheres to a surface of the tantalate particles; a form in which molybdenum replaces a portion of the crystal structure of the tantalate particles; an amorphous form; and a combination of any of these forms.

[0018]

In this specification, "the particle shape of the tantalate particles is controlled" means that the particle shape of the produced tantalate particles is not shapeless or irregular. In this specification, "tantalate particles having a controlled particle shape" means tantalate particles having a particle shape that is not shapeless or irregular.

[0019]

The tantalate particles of the embodiment may include at least one type of particles selected from the group consisting of $\text{K}_x\text{Na}_{(1-x)}\text{TaO}_3$ particles, where $x = 0$ to 1 ; $\text{Na}_2\text{Ta}_4\text{O}_{11}$ particles; and $\text{K}_2\text{Ta}_4\text{O}_{11}$ particles.

[0020]

A type and a composition of the tantalate salt present in the tantalate particles of the embodiment can be identified from an XRD spectrum pattern obtained by XRD analysis.

[0021]

The production method according to an embodiment, which will be described later, provides excellent control of the shape of the tantalate particles that are to be produced, with the control being achieved in accordance with a

crystal shape associated with any of the possible compositions. The tantalate particles of the embodiment have a controlled crystal shape and can have a distinct euohedral shape, such as a cubic shape, a polyhedral shape, or a columnar shape. Tantalate particles having any of these shapes can be produced by using the production method described later.

[0022]

The tantalate particles of the embodiment may include at least one type of particles selected from the group consisting of $K_xNa_{(1-x)}TaO_3$ particles having a cubic shape, where $x = 0$ to 1 ; $Na_2Ta_4O_{11}$ particles having a polyhedral shape; and $K_2Ta_4O_{11}$ particles having a polyhedral shape.

[0023]

In this specification, the "cubic shape" may be a shape derived from a perovskite structure; preferably, the shape may be a hexahedral shape that is rectangular or regular parallelepiped, and each of the faces that constitute the hexahedron may be a planar face, a curved face, or a face with irregularities. In this specification, preferably, the "polyhedral shape" is a shape having more than six faces, and the shapes of the faces may be different from one another. Each of the faces may be a planar face, a curved face, or a face with irregularities. In this specification, the "columnar shape" encompasses prismatic columnar shapes, cylindrical columnar shapes, rod shapes, and the like. A shape of a bottom face of the columnar body of the columnar tantalate particles is not particularly limited. Examples of the shape include circular shapes, elliptical shapes, and polygonal shapes. The columnar shape encompasses shapes that extend linearly in a longitudinal direction, shapes that extend obliquely, shapes that extend in a curved manner, shapes that extend in a branched manner, and the like. The columnar shape is a shape having an aspect ratio of greater than or equal to 2, where the aspect ratio is a ratio of a long diameter to

a short diameter.

[0024]

In instances where the tantalate particles include $K_xNa_{(1-x)}TaO_3$ particles having a cubic shape, it is preferable that the $K_xNa_{(1-x)}TaO_3$ particles have a particle size of 0.1 to 100 μm . The particle size is more preferably 0.5 to 50 μm and even more preferably 1 to 40 μm .

[0025]

In this specification, the "particle size" of the tantalate particles having a cubic shape is a length of an edge of a hexahedron identified in a particle image of primary particles of the tantalate particles, and the particle image is an image in a two-dimensional image captured with a scanning electron microscope (SEM). The value of the particle size of the tantalate particles having a cubic shape is an average value of the particle sizes of 50 or more tantalate particles, which are randomly selected from particles having a euohedral shape that is the target for the measurement.

[0026]

In instances where tantalate particles having a cubic shape are included, it is preferable that 50% or more of all the particles, on a weight or number basis, have a cubic shape; the percentage of particles having a cubic shape is more preferably 80% or more and even more preferably 90% or more.

[0027]

As an amount of use of molybdenum relative to that of tantalum increases, and a firing temperature increases, there is an increased tendency for $K_xNa_{(1-x)}TaO_3$ particles having a large particle size value to be produced.

[0028]

In instances where the tantalate particles include $Na_2Ta_4O_{11}$ particles having a polyhedral shape or $K_2Ta_4O_{11}$ particles having a polyhedral shape, it is preferable that the $Na_2Ta_4O_{11}$ particles or the $K_2Ta_4O_{11}$ particles have a particle

size of 1 to 1000 μm . The particle size is more preferably 2 to 500 μm , even more preferably 3 to 250 μm , and particularly preferably 3 to 100 μm .

[0029]

In this specification, the "particle size" of the $\text{Na}_2\text{Ta}_4\text{O}_{11}$ particles having a polyhedral shape and the $\text{K}_2\text{Ta}_4\text{O}_{11}$ particles having a polyhedral shape is a length of a maximum point-to-point distance, with the points being points on an exterior contour of a primary particle in a particle image of the tantalate particles, and the particle image is an image in a two-dimensional image captured with a scanning electron microscope (SEM). The value of the particle size is an average value of the particle sizes of 50 or more tantalate particles, which are randomly selected from particles having a euohedral shape that is the target for the measurement.

[0030]

In instances where tantalate particles having a polyhedral shape are included, it is preferable that 50% or more of all the particles, on a weight or number basis, have a polyhedral shape; the percentage of particles having a polyhedral shape is more preferably 80% or more and even more preferably 90% or more.

[0031]

As the amount of use of molybdenum relative to that of tantalum increases, and the firing temperature increases, there is an increased tendency for $\text{Na}_2\text{Ta}_4\text{O}_{11}$ particles or $\text{K}_2\text{Ta}_4\text{O}_{11}$ particles having a large particle size value to be produced.

[0032]

The particles of the embodiment may be particles produced by the method of a later-described embodiment for producing tantalate particles, with the particles containing molybdenum and exhibiting any of the XRD spectrum patterns shown in Fig. 16 and Fig. 17 (which are specifically the XRD spectrum patterns denoted as Example 9, Example 10, and

Example 11, excluding the XRD spectrum patterns of KTaO_3 and $\text{K}_2\text{Ta}_4\text{O}_{11}$, which are references). The XRD spectra are those obtained by performing a measurement under the following conditions: Cu-K α radiation, 40 kV/40 mA, a scan speed of 2°/min, and a scanning range of 10 to 90°.

[0033]

More specifically, the particles of the embodiment may be particles produced by a production method that includes firing a tantalum compound in the presence of a molybdenum compound and at least one of a potassium compound and a sodium compound, with the particles containing molybdenum and exhibiting any of the XRD spectrum patterns shown in Fig. 16 and Fig. 17.

[0034]

The XRD spectrum pattern shown in Fig. 16 may be determined to have peaks at $2\theta = 16.0 \pm 1.0^\circ$, $22.0 \pm 1.0^\circ$, $26.0 \pm 1.0^\circ$, $32.0 \pm 1.0^\circ$, and $46.0 \pm 1.0^\circ$.

[0035]

The XRD spectrum patterns shown in Fig. 17 may be determined to have peaks at $2\theta = 16.0 \pm 1.0^\circ$, $22.0 \pm 1.0^\circ$, $26.0 \pm 1.0^\circ$, $28.5 \pm 1.0^\circ$, $29.0 \pm 1.0^\circ$, $32.0 \pm 1.0^\circ$, and $52.0 \pm 1.0^\circ$.

[0036]

In another aspect, the particles of the embodiment may be particles produced by a production method that includes firing a tantalum compound in the presence of a molybdenum compound and at least one of a potassium compound and a sodium compound, with the particles containing molybdenum, exhibiting any of the XRD spectrum patterns shown in Fig. 16 and Fig. 17, and having a columnar shape.

[0037]

In instances where the particles that exhibit any of the XRD spectrum patterns shown in Fig. 16 and Fig. 17 include particles having a columnar shape, the particles may be, for example, particles having a long diameter of 1 to 2000 μm , a short diameter of 0.05 to 1000 μm , and an aspect ratio of 2 to 100, where the aspect ratio is a ratio of the

long diameter to the short diameter.

[0038]

In instances where the particles include particles having a columnar shape, it is preferable that the particles have a long diameter of 1 to 2000 μm ; the long diameter is more preferably 5 to 1000 μm and even more preferably 50 to 500 μm .

[0039]

In instances where the particles include particles having a columnar shape, it is preferable that the particles have a short diameter of 0.05 to 1000 μm ; the short diameter is more preferably 0.1 to 500 μm and even more preferably 10 to 250 μm .

[0040]

It is preferable that the particles having a columnar shape have an aspect ratio of greater than or equal to 2, where the aspect ratio is a ratio of the long diameter to the short diameter. The aspect ratio is more preferably 2 to 100, even more preferably 3 to 100, and still more preferably 5 to 50.

[0041]

In this specification, the "long diameter" of particles is a length of a long side of a bounding rectangle that surrounds a particle image of a primary particle of the particles (the bounding rectangle is defined such that the rectangle has a minimum area), and the particle image is an image in a two-dimensional image captured with a scanning electron microscope (SEM). In this specification, the "short diameter" of particles is a length represented by a straight line that connects two farthest points on the outer periphery of the particle image, in a direction perpendicular to the long diameter. Note that in the instance where the particles have a columnar shape, if the particles are compared to a fiber, the long diameter generally corresponds to a length of the fiber, and the short diameter generally corresponds to a diameter of the

fiber, and, in some cases, depending on the arrangement of the particles captured as an image, a deviation between the values of the actual long diameter and short diameter of a particle and the values of the measurements obtained from the two-dimensional image may be large. Accordingly, in instances where a representative shape of sample particles is recognized to be a columnar shape, particles that exhibit a face parallel to a longitudinal direction thereof in the captured image are to be appropriately selected and used as the target for the measurement. The values of the long diameter and the short diameter are average values of the long diameters and the short diameters of 50 or more particles, which are randomly selected from particles having a euhedral shape that is the target for the measurement.

[0042]

In instances where particles having a columnar shape are included, it is preferable that 50% or more of all the particles, on a weight or number basis, have a columnar shape; the percentage of particles having a columnar shape is more preferably 80% or more and even more preferably 90% or more.

[0043]

As the firing temperature increases while an excessive increase in the amount of use of molybdenum relative to that of tantalum is avoided, there is an increased tendency for particles having large values of the long diameter and the short diameter to be produced.

[0044]

A content of molybdenum in the tantalate particles of the embodiment, as determined by XRF analysis of the tantalate particles, may be greater than or equal to 0.01 mass%, 0.01 to 20 mass%, 0.05 to 15 mass%, or 0.1 to 10 mass%, in terms of a content percentage calculated as MoO_3 , relative to a total mass of the tantalate particles taken as 100 mass%.

[0045]

The content percentage calculated as MoO_3 is a value determined from an amount of MoO_3 calculated from a content of molybdenum determined by XRF analysis, and the calculation is carried out by using a MoO_3 -based calibration curve.

[0046]

A content of tantalum in the tantalate particles of the embodiment, as determined by XRF analysis of the tantalate particles, may be greater than or equal to 50 mass%, 50 to 99 mass%, 60 to 98 mass%, or 70 to 96 mass%, in terms of a content percentage calculated as Ta_2O_5 , relative to the total mass of the tantalate particles taken as 100 mass%.

[0047]

The content percentage calculated as Ta_2O_5 is a value determined from an amount of Ta_2O_5 calculated from a content of tantalum determined by XRF analysis, and the calculation is carried out by using a Ta_2O_5 -based calibration curve.

[0048]

A content of at least one of potassium and sodium in the tantalate particles, as determined by XRF analysis of the tantalate particles, may be greater than or equal to 0.5 mass%, 0.5 to 40 mass%, 1 to 30 mass%, or 2 to 20 mass%, in terms of a total content percentage calculated as at least one of K_2O and Na_2O , relative to the total mass of the tantalate particles taken as 100 mass%.

[0049]

The total content percentage calculated as at least one of K_2O and Na_2O is a value determined from a sum of an amount of K_2O and an amount of Na_2O calculated from a content of potassium determined by XRF analysis and a content of sodium determined by XRF analysis, and the calculation is carried out by using a K_2O -based calibration curve and a Na_2O -based calibration curve. Note that when the tantalate particles have a composition represented by $\text{K}_x\text{Na}_{(1-x)}\text{TaO}_3$, and $x = 0$ or 1 , the content of potassium or the content of

sodium may be zero.

[0050]

The values of the content of molybdenum, the content of tantalum, and the content of at least one of potassium and sodium may be selected and combined as desired. Examples of the tantalate particles of the embodiment include tantalate particles in which, as determined by XRF analysis of the tantalate particles, the content percentage of molybdenum calculated as MoO_3 is 0.01 to 20 mass%, the content percentage of tantalum calculated as Ta_2O_5 is 50 to 99 mass%, and the content percentage of at least one of potassium and sodium calculated as at least one of K_2O and Na_2O is 0.5 to 40 mass%, where these content percentages are percentages relative to the total mass of the tantalate particles taken as 100 mass%. Examples of the tantalate particles of the embodiment also include tantalate particles in which, as determined by XRF analysis of the tantalate particles, the content percentage of molybdenum calculated as MoO_3 is 0.05 to 15 mass%, the content percentage of tantalum calculated as Ta_2O_5 is 60 to 98 mass%, and the content percentage of at least one of potassium and sodium calculated as at least one of K_2O and Na_2O is 1 to 30 mass%, where these content percentages are percentages relative to the total mass of the tantalate particles taken as 100 mass%. Examples of the tantalate particles of the embodiment also include tantalate particles in which, as determined by XRF analysis of the tantalate particles, the content percentage of molybdenum calculated as MoO_3 is 0.1 to 10 mass%, the content percentage of tantalum calculated as Ta_2O_5 is 70 to 96 mass%, and the content percentage of at least one of potassium and sodium calculated as at least one of K_2O and Na_2O is 2 to 20 mass%, where these content percentages are percentages relative to the total mass of the tantalate particles taken as 100 mass%.

[0051]

The tantalate particles of the embodiment may be provided as an assembly of tantalate particles, and the values of the content of molybdenum, the content of tantalum, the content of potassium, and the content of sodium may be values determined from a sample of the assembly.

[0052]

In the tantalate particles of the embodiment, the tantalate salt, represented by $K_xNa_{(1-x)}Ta_yO_z$, may be present in an amount of greater than or equal to 50 mass%, 60 to 99.5 mass%, 65 to 99 mass%, or 70 to 95 mass%, relative to the total mass of the tantalate particles taken as 100 mass%.

[0053]

The tantalate particles of the embodiment can be produced, for example, by using the method for producing tantalate particles, which is described later. Note that the tantalate particles of the present invention are not limited to those produced by the method for producing tantalate particles of the embodiment described below.

[0054]

The tantalate particles of the embodiment have properties of both a tantalate salt and molybdenum and, therefore, are very useful.

[0055]

The tantalate particles of the embodiment can be used as piezoelectric materials, catalysts, semiconductor photoelectrodes, and the like.

[0056]

[Method for Producing Tantalate Particles]

According to an embodiment, a method for producing tantalate particles is a method for producing tantalate particles that includes firing a tantalum compound in the presence of a molybdenum compound and at least one of a potassium compound and a sodium compound.

[0057]

With the method for producing tantalate particles of the present embodiment, it is possible to produce the above-

described, molybdenum-containing tantalate particles of the embodiment of the present invention.

[0058]

Furthermore, in the method for producing tantalate particles of the present embodiment, the tantalum compound is fired in the presence of a molybdenum compound and at least one of a potassium compound and a sodium compound, and, consequently, the crystal shape of the tantalate particles that are to be produced can be readily controlled.

[0059]

Preferably, the method for producing tantalate particles includes a step of forming a mixture by mixing together a tantalum compound, a molybdenum compound, and at least one of a potassium compound and a sodium compound (mixing step) and a step of firing the mixture (firing step).

[0060]

Note that a compound containing molybdenum and potassium, such as potassium molybdate, may be used in place of at least a portion of the molybdenum compound and at least a portion of the potassium compound. Likewise, a compound containing molybdenum and sodium, such as sodium molybdate, may be used in place of at least a portion of the molybdenum compound and at least a portion of the sodium compound. Accordingly, a step of forming a mixture by mixing a tantalum compound with a compound containing molybdenum and at least one of potassium and sodium is considered to be equivalent to the step of forming a mixture by mixing together a tantalum compound, a molybdenum compound, and at least one of a potassium compound and a sodium compound.

[0061]

In instances where the tantalate particles that are to be produced contain potassium tantalate, the method for producing tantalate particles of the present embodiment may include a step of, prior to a firing step, forming a

mixture by mixing together a tantalum compound, a molybdenum compound, and a potassium compound (mixing step) and include a step of firing the mixture (firing step).

[0062]

In instances where the tantalate particles that are to be produced contain sodium tantalate, the method for producing tantalate particles of the present embodiment may include a step of, prior to a firing step, forming a mixture by mixing together a tantalum compound, a molybdenum compound, and a sodium compound (mixing step) and include a step of firing the mixture (firing step).

[0063]

[Mixing Step]

The mixing step is a step of forming a mixture by mixing together a tantalum compound, a molybdenum compound, and at least one of a potassium compound and a sodium compound. Details of the mixture will be described below.

[0064]

[Tantalum Compound]

The tantalum compound may be any compound provided that the compound can be fired together with one or more raw material compounds to form a tantalate salt. Examples of the tantalum compound include tantalum oxide, tantalum hydroxide, tantalum sulfide, tantalum nitride, tantalum halides, such as tantalum fluoride, tantalum chloride, tantalum bromide, and tantalum iodide, and tantalum alkoxide. Preferably, the tantalum compound is tantalum hydroxide or tantalum oxide, and more preferably, tantalum oxide. Examples of the tantalum oxide include tantalum pentoxide (Ta_2O_5), tantalum dioxide (TaO_2), and tantalum monoxide (TaO). One or more other tantalum oxides having a different valence may be used in addition to or in place of one or more of the tantalum oxides having the mentioned oxidation number. Physical properties of these tantalum compounds, which serve as precursors, are not particularly limited. Examples of the physical properties include

shapes, particle diameters, and specific surface areas.

[0065]

The tantalate particles after firing have a shape in which the shape of the tantalum compound as a raw material is substantially not shown. For example, the tantalate particles may have a shape, such as a spherical shape, an irregular shape, a shape of a structure having an aspect (e.g., a wire, a fiber, a ribbon, or a tube), or a sheet shape. Any of these shapes is suitable.

[0066]

[Molybdenum Compound]

Examples of the molybdenum compound include molybdenum oxide, molybdic acid, molybdenum sulfide, and molybdate compounds. Preferably, the molybdenum compound is molybdenum oxide or a molybdate compound.

[0067]

Examples of the molybdenum oxide include molybdenum dioxide (MoO_2) and molybdenum trioxide (MoO_3). Preferably, the molybdenum oxide is molybdenum trioxide.

[0068]

Preferably, the molybdate compound is an alkali metal salt of a molybdenum oxoanion. The molybdate compound is more preferably lithium molybdate, potassium molybdate, or sodium molybdate and even more preferably potassium molybdate or sodium molybdate.

[0069]

In the method for producing tantalate particles of the present embodiment, the molybdenum compound may be a hydrate.

[0070]

Preferably, the molybdenum compound is at least one compound selected from the group consisting of molybdenum trioxide, lithium molybdate, potassium molybdate, and sodium molybdate. More preferably, the molybdenum compound is at least one compound selected from the group consisting of molybdenum trioxide, potassium molybdate, and sodium

molybdate.

[0071]

Compounds containing molybdenum and potassium are suitable as a flux agent. Such a compound can be produced, for example, in the process of firing raw materials of a molybdenum compound and a potassium compound, which are less expensive and are more readily available. In this specification, instances in which a molybdenum compound and a potassium compound are used as flux agents and instances in which a compound containing molybdenum and potassium is used as a flux agent are both considered to be equivalent to the instance in which a molybdenum compound and a potassium compound are used as flux agents, that is, the "presence of a molybdenum compound and a potassium compound".

[0072]

Compounds containing molybdenum and sodium are suitable as a flux agent. Such a compound can be produced, for example, in the process of firing raw materials of a molybdenum compound and a sodium compound, which are less expensive and are more readily available. In this specification, instances in which a molybdenum compound and a sodium compound are used as flux agents and instances in which a compound containing molybdenum and sodium is used as a flux agent are both considered to be equivalent to the instance in which a molybdenum compound and a sodium compound are used as flux agents, that is, the "presence of a molybdenum compound and a sodium compound".

[0073]

Note that the molybdenum compounds mentioned above may be used alone or in a combination of two or more.

[0074]

Furthermore, the potassium molybdate ($K_2Mo_nO_{3n+1}$, $n = 1$ to 3) contains potassium and, therefore, can have functions of the potassium compound, which will be described below.

[0075]

Furthermore, the sodium molybdate ($\text{Na}_2\text{Mo}_n\text{O}_{3n+1}$, $n = 1$ to 3) contains sodium and, therefore, can have functions of the sodium compound, which will be described below.

[0076]

[Potassium Compound]

Examples of the potassium compound include, but are not limited to, potassium chloride, potassium chlorite, potassium chlorate, potassium sulfate, potassium hydrogen sulfate, potassium sulfite, potassium bisulfite, potassium nitrate, potassium carbonate, potassium hydrogen carbonate, potassium acetate, potassium oxide, potassium bromide, potassium bromate, potassium hydroxide, potassium silicate, potassium phosphate, potassium hydrogen phosphate, potassium sulfide, potassium hydrogen sulfide, potassium molybdate, and potassium tungstate. Examples of the potassium compound include isomers, as with the molybdenum compound. Among these, potassium carbonate, potassium hydrogen carbonate, potassium oxide, potassium hydroxide, potassium chloride, potassium sulfate, and potassium molybdate are preferable. Potassium carbonate, potassium hydrogen carbonate, potassium chloride, potassium sulfate, and potassium molybdate are more preferable.

[0077]

Note that the potassium compounds mentioned above may be used alone or in a combination of two or more.

[0078]

Furthermore, as with what is described above, the potassium molybdate contains molybdenum and, therefore, can have functions of the molybdenum compound described above.

[0079]

[Sodium Compound]

Examples of the sodium compound include, but are not limited to, sodium carbonate, sodium molybdate, sodium oxide, sodium sulfate, sodium hydroxide, sodium nitrate, sodium chloride, and metallic sodium. Among these, sodium carbonate, sodium molybdate, sodium oxide, and sodium

sulfate are preferable in terms of industrially easy availability and ease of handling.

[0080]

Note that the sodium compound mentioned above may be used alone or in a combination of two or more.

[0081]

Furthermore, as with what is described above, the sodium molybdate contains molybdenum and, therefore, can have functions of the molybdenum compound described above.

[0082]

Thus, some of the compounds are redundantly referred to as a molybdenum compound when the compounds are thus classified. It is preferable that the above-described molybdenum compound be at least one compound selected from the group consisting of molybdenum trioxide, potassium molybdate, and sodium molybdate, the above-described sodium compound be sodium carbonate or sodium molybdate, and the above-described potassium compound be potassium carbonate or potassium molybdate.

[0083]

A preferred example of the method is a method for producing sodium tantalate particles that includes firing a tantalum compound in the presence of a molybdenum compound and a sodium compound.

[0084]

A preferred example of the method is a method for producing potassium tantalate particles that includes firing a tantalum compound in the presence of a molybdenum compound and a potassium compound.

[0085]

A preferred example of the method is a method for producing potassium sodium tantalate particles that includes firing a tantalum compound in the presence of a molybdenum compound, a sodium compound, and a potassium compound.

[0086]

A preferred example of the method is a method for producing

potassium tantalate particles that includes firing a tantalum compound in the presence of a compound containing molybdenum and potassium.

[0087]

A preferred example of the method is a method for producing sodium tantalate particles that includes firing a tantalum compound in the presence of a compound containing molybdenum and sodium.

[0088]

A preferred example of the method is a method for producing potassium sodium tantalate particles that includes firing a tantalum compound in the presence of a compound containing molybdenum, potassium, and sodium.

[0089]

In the method for producing tantalate particles of the present embodiment, the molybdenum compound is used as a flux agent. In this specification, the production method, which uses a molybdenum compound as a flux agent, may be hereinafter referred to simply as a "flux method". Note that the following assumption can be made. In the firing, a tantalum compound, a molybdenum compound, and a sodium compound or a potassium compound (instances in which a tantalum compound and a compound containing molybdenum and sodium are used and instances in which a tantalum compound and a compound containing molybdenum and potassium are used are also encompassed) react with one another at a high temperature, and, consequently, one or more molybdate compounds that serve as flux (e.g., $K_aMo_bO_c$, $Na_aMo_bO_c$, and $K_aNa_a'Mo_bO_c$) are formed, and tantalate particles ($K_xNa_{(1-x)}Ta_yO_z$) are formed. It can be assumed that during the formation (crystal growth) of the tantalate particles, a molybdenum compound is partially incorporated into the tantalate particles. More specifically, the mechanism by which a molybdenum compound to be included in the tantalate particles is formed can be assumed to be as follows. During firing, in the process of the crystal growth of the

tantalate particles, a molybdenum compound, such as molybdenum oxide, is formed via the formation and decomposition of Mo-O-Ta in the system or via a molybdate compound that serves as a flux agent. Furthermore, in view of the mechanism, it can also be assumed that molybdenum oxide exists on a surface of the tantalate particles via the linkage of Mo-O-Ta. The presence of a molybdenum compound (e.g., molybdenum oxide) in the tantalate particles leads to improvement in physical properties of the tantalate particles, for example, improvement in the catalytic performance of the tantalate particles.

[0090]

The molybdate compound that serves as flux does not vaporize even in a firing temperature range, and, therefore, can be readily recovered by performing washing after firing. As a result, an amount of the molybdenum compound released to the outside from a firing furnace is reduced, and production cost is significantly reduced.

[0091]

In the method for producing tantalate particles of the present embodiment, a total amount of use of the raw materials that are believed to serve as flux agents, namely, a molybdenum compound, a potassium compound, and a sodium compound (hereinafter also referred to as "flux agents") and an amount of use of a tantalum compound as a raw material are not particularly limited, and the total amount of use of the flux agents may preferably be greater than or equal to 10 parts by mass, more preferably 20 to 5000 parts by mass, and even more preferably 30 to 1000 parts by mass, per 100 parts by mass of the tantalum compound as a raw material; a mixture may be formed by mixing together such amounts of the raw materials, and the mixture may be fired. The total amount of use of the flux agents may be 40 to 400 parts by mass per 100 parts by mass of the tantalum compound, a mixture may be formed by mixing together such amounts of the raw materials, and the mixture

may be fired; this is particularly preferable because in this case, particles having a particle size of less than or equal to 500 μm can be easily obtained.

[0092]

From the standpoint of efficient crystal growth, it is preferable that the amount of use of the flux agents relative to the amount of use of the tantalum compound as a raw material be greater than or equal to the lower limit. When the amount of use of the flux agents relative to the amount of use of the tantalum compound as a raw material is increased, there is a tendency for the size of the tantalate particles that are to be produced to be easily controlled. From the standpoint of reducing the amount of the flux agents used and improving production efficiency, it is preferable that the amount of use of the flux agents be less than or equal to the upper limit.

[0093]

In the method for producing tantalate particles of the present embodiment, a molar ratio of molybdenum atoms to tantalum atoms in the mixture is preferably greater than or equal to 0.01, more preferably greater than or equal to 0.03, even more preferably greater than or equal to 0.05, and particularly preferably greater than or equal to 0.1. The upper limit of the molar ratio of molybdenum atoms to tantalum atoms in the mixture may be appropriately specified. From the standpoint of reducing the amount of the molybdenum compound used and improving production efficiency, the molar ratio may be specified to be less than or equal to 5, less than or equal to 3, less than or equal to 2, or less than or equal to 1.5, for example. Examples of the numerical ranges of the molar ratio of molybdenum atoms to tantalum atoms in the mixture are as follows: the molar ratio is preferably 0.01 to 5, more preferably 0.03 to 3, even more preferably 0.05 to 2, and particularly preferably 0.1 to 1.5.

[0094]

When the molar ratio of molybdenum atoms to tantalum atoms is within any of the above-mentioned ranges, tantalate particles having a distinct euhedral shape, such as a cubic shape, a polyhedral shape, or a columnar shape, can be readily obtained, and, therefore, such ranges are preferable.

[0095]

In the method for producing tantalate particles of the present embodiment, a molar ratio of at least one of potassium atoms and sodium atoms to molybdenum atoms in the mixture is preferably 1 to 5 and more preferably 2 to 4.

In particular, when the molar ratio of at least one of potassium atoms and sodium atoms to molybdenum atoms is 2 to 4, a single composition of $K_xNa_{(1-x)}Ta_yO_z$ can be readily formed in the tantalate particles that are to be produced.

[0096]

In the method for producing tantalate particles of the present embodiment, a ratio of K to Na in the tantalate particles that are to be produced can be varied in accordance with a mixing ratio of K to Na in the raw materials. A molar ratio of at least one of potassium atoms and sodium atoms to molybdenum atoms in the mixture may be appropriately selected in accordance with a desired value of x and a desired value of $1-x$, which are values in $K_xNa_{(1-x)}Ta_yO_z$, for the tantalate particles that are to be produced. When $0 < x < 1$, a molar ratio of potassium atoms to sodium atoms in the mixture may be 0.01 to 10 or 0.1 to 5, for example.

[0097]

In instances where the compounds are used within any of the above-mentioned ranges, an amount of the molybdenum compound present in the resulting tantalate particles becomes more appropriate, and in addition, tantalate particles having a controlled crystal shape can be readily obtained.

[0098]

[Firing Step]

The firing step is a step of firing the mixture. The tantalate particles of the embodiment can be obtained by firing the mixture. As stated above, this production method is referred to as a flux method.

[0099]

The flux method is classified as a solution method. More specifically, the flux method is a method for growing crystals that utilizes an instance in which eutectic properties are exhibited in a crystal-flux binary phase diagram. It is speculated that the mechanism of the flux method is as follows. Specifically, as a mixture of a solute and flux is heated, the solute and the flux form a liquid phase. In this case, since the flux is a fusing agent, that is, eutectic properties are exhibited in a solute-flux binary phase diagram, the solute melts at a temperature lower than its melting point to form the liquid phase. When the flux is evaporated in this state, the concentration of the flux decreases, that is, the effect of the flux of decreasing the melting point of the solute is reduced, and thus, the evaporation of the flux serves as a driving force to cause the crystal growth of the solute (flux evaporation method). Note that the crystal growth of the solute can also be caused by cooling the liquid phase of the solute and the flux (slow cooling method).

[0100]

The flux method has advantages. For example, crystal growth can be achieved at temperatures much lower than a melting point, crystal structures can be precisely controlled, and a crystal body having a euhedral shape can be formed.

[0101]

The mechanism by which tantalate particles are produced by a flux method that uses a molybdenum compound as flux is not necessarily clear. However, for example, it is speculated that the mechanism is as follows. Specifically,

when a tantalum compound is fired in the presence of a molybdenum compound, a portion of the tantalum compound forms tantalum molybdate, and the molybdenum compound forms one or more molybdate salts (e.g., $K_aMo_bO_c$, $Na_aMo_bO_c$, and $K_aNa_a'Mo_bO_c$). In this case, a fluxing function of the molybdate salt enables the crystal of a tantalate salt to grow at a temperature lower than the melting point of the tantalate salt, as will be appreciated from the description above. Furthermore, for example, the tantalum molybdate derived from a portion of the tantalum compound is decomposed, and, accordingly, the crystal growth of the tantalate particles is promoted. That is, the molybdenum compound (molybdate salt) serves as flux, and, via the tantalum molybdate, which is an intermediate product, the tantalate particles are produced.

[0102]

Methods for the firing are not particularly limited, and the firing may be performed by using a common method known in the art. It can be assumed that when a firing temperature exceeds 500°C, a portion of the tantalum compound reacts with the molybdenum compound to form tantalum molybdate, and the molybdenum compound forms one or more molybdate salts (e.g., $K_aMo_bO_c$, $Na_aMo_bO_c$, and $K_aNa_a'Mo_bO_c$). Furthermore, when the firing temperature reaches 800°C or greater, the tantalum molybdate derived from a portion of the tantalum compound is decomposed, and the tantalate particles are formed as a result of the fluxing function of the molybdate salt. Furthermore, in the case of tantalate particles, it can be assumed that a molybdenum compound is incorporated into the tantalate particles during the processes of the decomposition of the tantalum molybdate and the crystal growth of the particles.

[0103]

Furthermore, the states of the tantalum compound, the molybdenum compound, the sodium compound, the potassium compound, and the like that can be used in the firing are

not particularly limited, and it is sufficient that the raw material compounds, namely, the molybdenum compound, the tantalum compound, the sodium compound, the potassium compound, and the like be present in the same space in which the raw material compounds can act on one another. Specifically, any of the following mixing methods may be employed: simple mixing in which powders of the raw material compounds are mixed together, mechanical mixing using a mill or the like, and mixing using a mortar or the like; and either of dry mixing and wet mixing may be employed.

[0104]

The conditions of the firing temperature are not particularly limited and are appropriately determined in consideration of the particle size of the target tantalate particles, the formation of a molybdenum compound in the tantalate particles, the shape of the tantalate particles, and the like. The firing temperature may be greater than or equal to 700°C, which is close to the temperature at which a molybdate salt can serve as flux. The firing temperature may be greater than or equal to 750°C, greater than or equal to 800°C, greater than or equal to 850°C, or greater than or equal to 900°C. From the standpoint of efficiently producing tantalate particles having a distinct euhedral shape, such as a cubic shape, a polyhedral shape, or a columnar shape, it is preferable that the firing temperature be greater than or equal to 800°C. The firing temperature is more preferably greater than or equal to 900°C and even more preferably greater than or equal to 1000°C.

[0105]

According to the results obtained in Examples, which will be described later, when the firing temperature is greater than or equal to 1100°C, $K_2Ta_4O_{11}$ particles or $Na_2Ta_4O_{11}$ particles can be easily obtained.

[0106]

In general, controlling the shape of the tantalate particles that result from firing requires the implementation of high-temperature firing at greater than 1500°C, which is close to the melting point of tantalum oxide. However, industrial application of such high-temperature firing involves significant problems in terms of the load on the firing furnace and the fuel cost.

[0107]

With an embodiment of the present invention, the formation of tantalate particles can be achieved efficiently and at low cost even under conditions in which, for example, a maximum firing temperature for firing the tantalum compound is less than or equal to 1500°C. Furthermore, with the method for producing tantalate particles of the present embodiment, it is possible to form tantalate particles having a euhedral shape regardless of the shape of the precursors, even at a firing temperature of less than or equal to 1300°C, which is a temperature much lower than the melting point of tantalum oxide. Furthermore, from the standpoint of efficiently producing the tantalate particles, the firing temperature may be specified to be less than or equal to 1200°C or less than or equal to 1100°C.

[0108]

Examples of the numerical ranges of the firing temperature for firing the tantalum compound in the firing step may include 700 to 1500°C, 750 to 1400°C, 800 to 1300°C, 850 to 1200°C, and 900 to 1100°C.

[0109]

In another aspect, examples of the numerical ranges of the firing temperature for firing the tantalum compound may include 700 to 1500°C, 750 to 1400°C, 800 to 1300°C, 850 to 1300°C, 900 to 1300°C, and 1000 to 1300°C.

[0110]

A heating rate may be specified to be 20 to 600°C/h, 40 to 500°C/h, 100 to 400°C/h, or 200 to 400°C/h, from the

standpoint of production efficiency. With the method for producing tantalate particles of the present embodiment, tantalate particles having a controlled crystal shape can be readily produced even at a high heating rate of greater than or equal to 100°C/h, because a molybdenum compound is used as flux.

[0111]

Regarding the time of firing, it is preferable that the heating time for increasing the temperature to a predetermined firing temperature be within a range of 15 minutes to 10 hours, and a holding time for holding at the firing temperature be within a range of 5 minutes to 30 hours. In terms of achieving efficient formation of the tantalate particles, it is preferable that the holding time associated with the firing temperature be greater than or equal to 2 hours, and it is more preferable that the holding time associated with the firing temperature be 2 to 15 hours. By selecting the conditions of a firing temperature of 700 to 1500°C and a holding time associated with the firing temperature of 2 to 15 hours, tantalate particles containing molybdenum and having a euhedral shape can be readily obtained. By selecting the conditions of a firing temperature of 900 to 1500°C and a holding time associated with the firing temperature of 2 to 15 hours, tantalate particles containing molybdenum and having a distinct euhedral shape, such as a cubic shape, a polyhedral shape, or a columnar shape, can be readily obtained.

[0112]

Atmospheres for the firing are not particularly limited provided that the effects of the present invention can be produced. For example, an oxygen-containing atmosphere, such as air or oxygen, or an inert atmosphere, such as nitrogen, argon, or carbon dioxide, is preferable, and, when cost is taken into consideration, an air atmosphere is more preferable.

[0113]

Apparatuses for performing the firing are also not necessarily limited, and a so-called firing furnace may be used. It is preferable that the firing furnace be formed of a material that does not react with sublimed molybdenum oxide, and it is further preferable that a gas-tight firing furnace be used to efficiently utilize the molybdenum oxide.

[0114]

[Cooling Step]

The method for producing tantalate particles may include a cooling step. The cooling step is a step of cooling the tantalate particles that have undergone crystal growth in the firing step.

[0115]

A cooling rate is not particularly limited and is preferably 1 to 1000°C/hour, more preferably 5 to 500°C/hour, even more preferably 100 to 400°C/hour, and particularly preferably 200 to 400°C/hour. When the cooling rate is greater than or equal to 1°C/hour, a production time can be shortened, and, accordingly, such a cooling rate is preferable. On the other hand, when the cooling rate is less than or equal to 1000°C/hour, cracking of a firing crucible due to heat shock does not occur frequently, that is, the firing crucible can be used for a long time, and, accordingly, such a cooling rate is preferable. With the method for producing tantalate particles of the present embodiment, tantalate particles having a controlled crystal shape can be readily produced even at a high cooling rate of greater than or equal to 200°C/hour, because a molybdenum compound is used as flux.

[0116]

Methods for the cooling are not particularly limited. Natural cooling may be employed, or a cooling device may be used.

[0117]

[Aftertreatment Step]

The production method of the present embodiment may include an aftertreatment step. The aftertreatment step may be a step of separating the tantalate particles from the flux agent, both included in the fired product, and the separating may be performed after the fired product is removed from the firing crucible. The aftertreatment step may be performed after the firing step described above. Furthermore, the aftertreatment step may be repeatedly performed, twice or more, as necessary.

[0118]

Examples of methods for removing the flux agent include washing and high-temperature treatment. These may be performed in combination.

[0119]

Methods for the washing are not particularly limited. In instances where the flux is water-soluble, as is the molybdate compound, examples of the methods include water washing.

[0120]

Furthermore, examples of methods for the high-temperature treatment include a method in which the fired product is heated to a temperature greater than or equal to the sublimation point or the boiling point of the flux.

[0121]

[Pulverizing Step]

In some cases, the fired product resulting from the firing step may include aggregates of tantalate particles, and, consequently, a particle diameter range suitable for applications that are being considered may not be satisfied. Accordingly, if necessary, the tantalate particles may be pulverized so that a suitable particle diameter range can be satisfied. Methods for pulverizing the fired product are not particularly limited. Any known pulverizing method using a ball mill, jaw crusher, jet mill, disc mill, SpectroMill, grinder, mixer mill, or the

like may be employed.

[0122]

[Size Classification Step]

The fired product resulting from the firing step and including the tantalate particles may be subjected to a size classification process when appropriate, so that the particle size range can be adjusted. The term "size classification process" refers to an operation of sorting particles by particle size. The size classification may be wet classification or dry classification, but, from the standpoint of productivity, dry classification is preferable. The dry classification may be classification using a sieve or may be, for example, air classification, in which classification is performed by using the difference between the centrifugal force and the fluid drag. From the standpoint of classification accuracy, air classification is preferable, and the air classification may be performed by using a classifier, such as an air sifter that utilizes a Coanda effect, a swirling airflow type classifier, a forced vortex centrifugal classifier, or a semi-free vortex centrifugal classifier. The pulverizing step and the size classification step described above may be performed at stages where the steps are necessary. By selecting whether or not to perform the pulverizing and/or the size classification and/or selecting conditions for the steps, the average particle diameter of the resulting tantalate particles, for example, can be adjusted.

[0123]

It is preferable that the tantalate particles of the embodiment or the tantalate particles produced by the production method of the embodiment have few aggregates or no aggregates. This is because in such a case, their inherent properties can be easily exhibited, and the handleability thereof is enhanced, and enhanced dispersibility is exhibited in an instance in which the tantalate particles are used by being dispersed in a

dispersion medium.

[0124]

Note that with the method for producing tantalate particles of the embodiment, tantalate particles having few aggregates or no aggregates can be readily produced; therefore, the method has an excellent advantage of producing, with high productivity, tantalate particles having desired excellent properties, without performing the pulverizing step or the size classification step.

[EXAMPLES]

[0125]

The present invention will now be described in more detail with reference to examples. It should be noted that the present invention is not limited to the examples described below.

[0126]

[Analysis and Evaluation]

The following measurements were performed on samples, which were powders of Examples and Comparative Examples.

[0127]

[Measurement of Particle Size]

[Instance of Particles Having Cubic Shape]

The sample powders were photographed with a scanning electron microscope (SEM). In an instance where a smallest unit particle (i.e., a primary particle), as seen in a two-dimensional image, was recognized to have a cubic shape, the length of an edge of a hexahedron identified in a particle image of the primary particle was measured and designated as the particle size. The same operation was performed on 50 primary particles, and an average value was determined for each of the samples.

[0128]

[Instance of Particles Having Columnar Shape]

The sample powders were photographed with a scanning electron microscope (SEM). In an instance where a smallest unit particle (i.e., a primary particle), as seen in a two-

dimensional image, was recognized to have a columnar shape, the long diameter and the short diameter of the particle were measured. The long diameter was defined as the length of a long side of a bounding rectangle that surrounds a particle image (the bounding rectangle was defined such that the rectangle has a minimum area). The short diameter was defined as the length represented by a straight line that connected two farthest points on the outer periphery of the particle image, in a direction perpendicular to the long diameter. Note that in the instance where a representative shape of the particles of a sample was recognized to be a columnar shape, particles that exhibited a face parallel to a longitudinal direction thereof in the captured image were appropriately selected and used as the target for the measurement. The same operation was performed on 50 primary particles, and an average value was determined for each of the samples. Furthermore, the ratio of the long diameter to the short diameter (long diameter/short diameter) was determined and designated as the aspect ratio of primary particles.

[0129]

[Instance of Particles Having Polyhedral Shape]

The sample powders were photographed with a scanning electron microscope (SEM). In an instance where a smallest unit particle (i.e., a primary particle), as seen in a two-dimensional image, was recognized to have a polyhedral shape, the length of a maximum point-to-point distance was measured and designated as the particle size, where the points were points on an exterior contour identified in a particle image of the primary particle. The same operation was performed on 50 primary particles, and an average value was determined for each of the samples.

[0130]

[Analysis of Crystal Structure: XRD (X-ray diffraction) Method]

The sample powder was loaded into a measurement sample

holder having a depth of 0.5 mm, which was then placed in a wide-angle X-ray diffractometer (XRD) (Ultima IV, manufactured by Rigaku Corporation), and a measurement was performed under the following conditions: Cu-K α radiation, 40 kV/40 mA, a scan speed of 2°/minute, and a scanning range of 10 to 70° or a scanning range of 10 to 90°.

[0131]

[XRF (X-ray fluorescence) Analysis]

Approximately 70 mg of the sample powder was placed on filter paper and covered with a PP film and then subjected to an XRF (X-ray fluorescence) analysis, which was performed by using an X-ray fluorescence spectrometer PrimusIV (manufactured by Rigaku Corporation). The conditions for the analysis were as follows.

Measurement conditions

EZ scan mode

Measured elements: F to U

Measurement time: standard

Measurement diameter: 10 mm

Residue component (balance component): none

[0132]

XRF analysis was performed on the sample powder to determine a content of molybdenum of the sample powder, and the content percentage (mass%) calculated as MoO₃, relative to the total mass of the sample powder taken as 100 mass%, was calculated. XRF analysis was performed on the sample powder to determine a content of tantalum of the sample powder, and the content percentage (mass%) calculated as Ta₂O₅, relative to the total mass of the sample powder taken as 100 mass%, was calculated. XRF analysis was performed on the sample powder to determine a content of potassium of the sample powder, and the content percentage (mass%) calculated as K₂O, relative to the total mass of the sample powder taken as 100 mass%, was calculated. XRF analysis was performed on the sample powder to determine a content of sodium of the sample powder, and the content

percentage (mass%) calculated as Na_2O , relative to the total mass of the sample powder taken as 100 mass%, was calculated.

[0133]

(1. Production of Tantalate Particles Having Cubic Shape)

[Example 1]

A mixture was obtained by mixing 10.0 g of tantalum oxide (Ta_2O_5 , a reagent manufactured by Kanto Chemical Co., Inc.) with 10.0 g of sodium molybdate dihydrate ($\text{Na}_2\text{MoO}_4 \cdot 2\text{H}_2\text{O}$, a reagent manufactured by Kanto Chemical Co., Inc.) in a mortar. The obtained mixture was placed in a crucible and then, in a ceramic electric furnace, heated at a heating rate of $300^\circ\text{C}/\text{h}$ to 1500°C and fired at 1500°C for 24 hours. Next, the mixture was cooled at a cooling rate of $300^\circ\text{C}/\text{h}$ to 500°C and thereafter naturally cooled to room temperature. Subsequently, the crucible was removed from the ceramic electric furnace. Subsequently, the obtained fired product was washed with water five times, and thereafter, the resultant was washed with water by filtration to remove the waste washing liquid and then dried. Thus, the remaining flux agent was removed, and, accordingly, a powder of Example 1 (9.4 g) was obtained.

[0134]

The synthesis conditions are shown in Table 1.

[0135]

[Example 2]

A powder of Example 2 was obtained by performing the same operation as that of Example 1, except that the amounts of use of the raw material compounds and the firing conditions were changed from those of Example 1 as shown in Table 1.

[0136]

[Example 3]

A powder of Example 3 was obtained by performing the same operation as that of Example 1, except that the amount of use of the tantalum oxide was changed as shown in Table 1, 3.87 g of molybdenum oxide (MoO_3 , Nippon Inorganic Colour &

Chemical Co., Ltd.) and 5.7 g of sodium carbonate (Na_2CO_3 , a reagent manufactured by Kanto Chemical Co., Inc.) were used instead of sodium molybdate, and the firing conditions were changed as shown in Table 1; these conditions were changed from those of Example 1.

[0137]

[Example 4]

A powder of Example 4 was obtained by performing the same operation as that of Example 1, except that 10.0 g of tantalum oxide, 6.8 g of molybdenum oxide, 5.0 g of sodium carbonate, and 6.53 g of potassium carbonate (K_2CO_3 , a reagent manufactured by Kanto Chemical Co., Inc.) were used as the raw material compounds, and the firing conditions were changed as shown in Table 1.

[0138]

(Examples 5 and 6)

Powders of Examples 5 and 6 were obtained by performing the same operation as that of Example 4, except that the amounts of use of the raw material compounds and the firing conditions were changed from those of Example 4 as shown in Table 1.

[0139]

(Comparative Example 1)

A powder of Comparative Example 1 was obtained by performing the same operation as that of Example 1, except that the amounts of use of the raw material compounds and the firing conditions were changed from those of Example 1 as shown in Table 1.

[0140]

[Results]

SEM images of the powders obtained in Examples 1 to 6 and Comparative Example 1, described above, are shown in Figs. 1 to 7.

[0141]

The result of each of the evaluations described above is shown in Table 1. The symbol "-" indicates that the

compound was not used. "N.D." is an abbreviation for "not detected" and means that the compound was not detected.

[0142]

[Table 1]

Producing condition										Evaluation								
	Ta ₂ O ₅	Na ₂ MoO ₄ ·2H ₂ O	MoO ₃	Na ₂ CO ₃	K ₂ CO ₃	amount of use of the flux agents relative to Ta ₂ O ₅	(K+Na)/Mo	K /Na	Mo /Ta	firing condition		SEM		XRF			XRD	
										temperature	holding time	shape	particle size	KxNa(1-x)Ta ₂ O ₅				
														Ta ₂ O ₅	K ₂ O	Na ₂ O		
																		mass %
	g	g	g	g	g	parts by mass	molar ratio	molar ratio	molar ratio	°C	h		μm	mass %	mass %	mass %	composition	
Example 1	10	10	-	-	-	100	2.0	-	0.9	1500	24	Cubic	35	91.6	N.D.	7.3	0.21	NaTaO ₃
Example 2	10	20	-	-	-	200	2.0	-	1.9	1300	24	Cubic	15	92.3	N.D.	5.52	1.79	NaTaO ₃
Example 3	6	-	3.87	5.7	-	160	4.0	-	1.0	1300	24	Cubic	8	93.1	N.D.	6.7	0.17	NaTaO ₃
Example 4	10	-	6.8	5	6.53	183	4.0	1.0	1.0	1100	10	Cubic	2	90.6	1.80	7.09	0.30	K _x Na _(1-x) TaO ₃
Example 5	10	-	6.8	5	6.53	183	4.0	1.0	1.0	900	10	Cubic	2	89.5	1.97	7.15	0.32	K _x Na _(1-x) TaO ₃
Example 6	10	-	6.8	7.5	4.03	183	4.2	0.4	1.0	900	10	Cubic	2	90.2	0.57	7.36	0.11	K _x Na _(1-x) TaO ₃
Comparative Example 1	10	-	-	-	-	0	-	-	-	900	10	Shapeless	<1	100	N.D.	N.D.	N.D.	Ta ₂ O ₅

[0143]

Table 1 shows the shape and the size of the particles of

the Examples and Comparative Example, which were determined from the SEM images. In instances where particles having different shapes were recognized to coexist, a representative shape (shape most frequently seen) is indicated. In instances where no particular shape was seen, a determination was made that the particles were shapeless or irregular.

[0144]

The results of the XRD analysis are shown in Figs. 8 and 9. In the samples of Examples 1 to 6, peaks were observed at $2\theta = 22.5 \pm 1.0^\circ$, $32.5 \pm 1.0^\circ$, and $46.5 \pm 1.0^\circ$, which were attributable to a perovskite crystal structure of NaTaO_3 or $\text{K}_x\text{Na}_{(1-x)}\text{TaO}_3$.

[0145]

The results of the SEM observation and the XRD analysis confirmed that the particles of the powders obtained in the Examples were tantalate particles containing $\text{K}_x\text{Na}_{(1-x)}\text{TaO}_3$ and having a cubic shape and a perovskite crystal structure.

[0146]

Furthermore, the results demonstrated that the tantalate particles of Examples 1 to 6 contained tantalum, potassium, sodium, and molybdenum in the amounts shown in Table 1, calculated as oxides, as determined by XRF analysis.

[0147]

The results of Examples 1 to 6 demonstrated that by firing a tantalum compound in the presence of a molybdenum compound and at least one of a potassium compound and a sodium compound, molybdenum-containing tantalate particles can be produced even at a relatively low firing temperature of 900 to 1500°C.

[0148]

Furthermore, the results demonstrated that by firing a tantalum compound in the presence of a molybdenum compound and at least one of a potassium compound and a sodium compound, the shape of the tantalate particles that are to

be produced can be readily controlled.

[0149]

(2. Production of Tantalate Particles Having Polyhedral Shape)

(Examples 7 and 8)

Powders of Examples 7 and 8 were obtained by performing the same operation as that of Example 1, except that the amounts of use of the raw material compounds and the firing conditions were changed from those of Example 1 as shown in Table 2.

[0150]

[Results]

SEM images of the powders obtained in Examples 7 and 8, described above, are shown in Figs. 10 and 11.

[0151]

The result of each of the evaluations described above is shown in Table 2. The symbol "-" indicates that the compound was not used. "N.D." is an abbreviation for "not detected" and means that the compound was not detected.

[0152]

[Table 2]

	Producing condition							Evaluation								
	Ta ₂ O ₅	Na ₂ MoO ₄ ·2H ₂ O	MoO ₃	K ₂ CO ₃	amount of use of the flux agents relative to Ta ₂ O ₅	(K+Na)/Mo	Mo/Ta	firing condition		SEM		XRF			XRD	
								temperature	holding time	shape	particle size	KxNa(1-x)TayOz				
												Ta ₂ O ₅	K ₂ O	Na ₂ O		
	g	g	g	g	parts by mass	molar ratio	molar ratio	°C	h		μm	mass %	mass %	mass %	composition	
Example 7	10	5	—	—	50	2	0.46	1300	24	polyhedral	20	95.4	N.D.	3.10	0.24	Na ₂ Ta ₄ O ₁₁
Example 8	10	—	5.1	4.9	100	2	0.78	1100	10	polyhedral	3	90.5	8.1	N.D.	1.2	K ₂ Ta ₄ O ₁₁

[0153]

Table 2 shows the shape and the size of the particles of

the Examples, which were determined from the SEM images. In instances where particles having different shapes were recognized to coexist, a representative shape (shape most frequently seen) is indicated.

[0154]

The results of the XRD analysis are shown in Fig. 12. In the samples of Examples 7 and 8, peaks attributable to $\text{Na}_2\text{Ta}_4\text{O}_{11}$ or $\text{K}_2\text{Ta}_4\text{O}_{11}$ were observed.

[0155]

The results of the SEM observation and the XRD analysis confirmed that the particles of the powder obtained in Example 7 were sodium tantalate particles containing $\text{Na}_2\text{Ta}_4\text{O}_{11}$ and having a polyhedral shape. The results confirmed that the particles of the powder obtained in Example 8 were potassium tantalate particles containing $\text{K}_2\text{Ta}_4\text{O}_{11}$ and having a polyhedral shape.

[0156]

Furthermore, the results demonstrated that the tantalate particles of Examples 7 and 8 contained tantalum, potassium, sodium, and molybdenum in the amounts shown in Table 2, calculated as oxides, as determined by XRF analysis.

[0157]

The results of Examples 7 and 8 demonstrated that by firing a tantalum compound in the presence of a molybdenum compound and one of a potassium compound and a sodium compound, molybdenum-containing tantalate particles can be produced even at a relatively low firing temperature of 1100 to 1300°C.

[0158]

Furthermore, the results demonstrated that by firing a tantalum compound in the presence of a molybdenum compound and one of a sodium compound and a potassium compound, the shape of the tantalate particles that are to be produced can be readily controlled.

[0159]

(3. Production of Tantalate Particles Having Columnar Shape)

(Examples 9 to 11)

Powders of Examples 9 to 11 were obtained by performing the same operation as that of Example 1, except that the amounts of use of the raw material compounds and the firing conditions were changed from those of Example 1 as shown in Table 3.

[0160]

[Results]

SEM images of the powders obtained in Examples 9 to 11, described above, are shown in Figs. 13 to 15.

[0161]

The result of each of the evaluations described above is shown in Table 3. The symbol "-" indicates that the compound was not used. "N.D." is an abbreviation for "not detected" and means that the compound was not detected.

[0162]

[Table 3]

	Producing condition							Evaluation								
	amount of use of the flux agents relative to Ta ₂ O ₅							firing condition		SEM				XRF		
	Ta ₂ O ₅	MoO ₃	Na ₂ CO ₃	K ₂ CO ₃	(K+Na)/Mo	Mo/Ta	temperature	holding time	shape	short diameter	long diameter	aspect ratio (long diameter/short diameter)	Ta ₂ O ₅	K ₂ O	Na ₂ O	MoO ₃
	g	g	g	g	parts by mass	molar ratio	°C	h		μm	μm		mass %	mass %	mass %	mass %
Example 9	6	5.9	2.26	—	136	1.0	1300	24	columnar	100	500	5	92.9	N.D.	2.2	0.5
Example 10	6	4.36	—	4.17	142	2.0	1300	24	columnar	40	200	5	88.7	6.07	N.D.	2.28
Example 11	6	5.43	—	2.59	134	1.0	1300	24	columnar	30	150	5	71.1	8.66	N.D.	18.5

[0163]

Table 3 shows the shape and the size of the particles of

Examples 9 to 11, which were determined from the SEM images. In instances where particles having different shapes were recognized to coexist, a representative shape (shape most frequently seen) is indicated.

[0164]

The results of the XRD analysis are shown in Figs. 16 and 17. It was difficult to identify the composition of the samples of Examples 9 to 11 from the results of the XRD analysis. The particles of Example 9 exhibited the peaks shown in Fig. 16; the peaks were observed at $2\theta = 16.0\pm 1.0^\circ$, $22.0\pm 1.0^\circ$, $26.0\pm 1.0^\circ$, $32.0\pm 1.0^\circ$, and $46.0\pm 1.0^\circ$. The particles of Examples 10 and 11 exhibited the respective peaks shown in Fig. 17; the peaks were observed at $2\theta = 16.0\pm 1.0^\circ$, $22.0\pm 1.0^\circ$, $26.0\pm 1.0^\circ$, $28.5\pm 1.0^\circ$, $29.0\pm 1.0^\circ$, $32.0\pm 1.0^\circ$, and $52.0\pm 1.0^\circ$.

[0165]

From the results of the SEM observation and the XRD analysis, it was inferred that the particles of the powders obtained in Examples 9 to 11 were tantalate particles having a columnar shape.

[0166]

Furthermore, the results demonstrated that the particles of Examples 9 to 11 contained tantalum, potassium, sodium, and molybdenum in the amounts shown in Table 3, calculated as oxides, as determined by XRF analysis.

[0167]

The results of Examples 9 to 11 demonstrated that by firing a tantalum compound in the presence of a molybdenum compound and one of a potassium compound and a sodium compound, particles containing molybdenum, tantalum, and one of potassium and sodium and having a columnar shape can be produced even at a relatively low firing temperature of 1300°C .

[0168]

Furthermore, the results demonstrated that the use of a molybdenum compound enables favorable crystal growth of the

particles, and, consequently, particles having a particle size of greater than or equal to 1 μm can be readily produced even under the firing conditions mentioned above.

[0169]

The tantalate particles of Examples 1 to 11 contain molybdenum and, therefore, are expected to exhibit various effects due to molybdenum, such as catalytic activity.

[0170]

The constituents, combinations thereof, and the like in the embodiments are merely illustrative. Addition, omission, and/or substitution of one or more constituents as well as other modifications may be made without departing from the spirit of the present invention. Furthermore, the present invention is not limited to the embodiments described but is only limited by the scope of the claims.

[CLAIMS]

[Claim 1]

Tantalate particles containing molybdenum and represented by $K_xNa_{(1-x)}Ta_yO_z$, where $x = 0$ to 1 , $y = 1$ to 10 , and $z = 3$ to 20 .

[Claim 2]

The tantalate particles according to Claim 1, wherein the tantalate particles include at least one type of particles selected from the group consisting of $K_xNa_{(1-x)}TaO_3$ particles, where $x = 0$ to 1 ; $Na_2Ta_4O_{11}$ particles; and $K_2Ta_4O_{11}$ particles.

[Claim 3]

The tantalate particles according to Claim 2, wherein the tantalate particles include at least one type of particles selected from the group consisting of $K_xNa_{(1-x)}TaO_3$ particles having a cubic shape, where $x = 0$ to 1 ; $Na_2Ta_4O_{11}$ particles having a polyhedral shape; and $K_2Ta_4O_{11}$ particles having a polyhedral shape.

[Claim 4]

The tantalate particles according to Claim 2 or 3, wherein the tantalate particles include the $K_xNa_{(1-x)}TaO_3$ particles, the $K_xNa_{(1-x)}TaO_3$ particles have a cubic shape, and the $K_xNa_{(1-x)}TaO_3$ particles have a particle size of 0.1 to $100\ \mu m$.

[Claim 5]

The tantalate particles according to Claim 2 or 3, wherein the tantalate particles include the $Na_2Ta_4O_{11}$ particles, the $Na_2Ta_4O_{11}$ particles have a polyhedral shape, and the $Na_2Ta_4O_{11}$ particles have a particle size of 1 to $1000\ \mu m$.

[Claim 6]

The tantalate particles according to Claim 2 or 3, wherein the tantalate particles include the $K_2Ta_4O_{11}$ particles, the $K_2Ta_4O_{11}$ particles have a polyhedral shape, and the $K_2Ta_4O_{11}$ particles have a particle size of 1 to $1000\ \mu m$.

[Claim 7]

The tantalate particles according to any one of Claims 1 to

6, wherein a content of the molybdenum in the tantalate particles, as determined by XRF analysis of the tantalate particles, is 0.01 to 20 mass% in terms of a content percentage calculated as MoO_3 , relative to a total mass of the tantalate particles taken as 100 mass%.

[Claim 8]

The tantalate particles according to any one of Claims 1 to 7, wherein a content of tantalum in the tantalate particles, as determined by XRF analysis of the tantalate particles, is 50 to 99 mass% in terms of a content percentage calculated as Ta_2O_5 , relative to a total mass of the tantalate particles taken as 100 mass%.

[Claim 9]

The tantalate particles according to any one of Claims 1 to 8, wherein a content of at least one of potassium and sodium in the tantalate particles, as determined by XRF analysis of the tantalate particles, is 0.5 to 40 mass% in terms of a total content percentage calculated as at least one of K_2O and Na_2O , relative to a total mass of the tantalate particles taken as 100 mass%.

[Claim 10]

A method for producing tantalate particles, the tantalate particles being the tantalate particles according to any one of Claims 1 to 9, the method comprising firing a tantalum compound in the presence of a molybdenum compound and at least one of a potassium compound and a sodium compound.

[Claim 11]

The method for producing tantalate particles according to Claim 10, wherein the molybdenum compound is at least one compound selected from the group consisting of molybdenum trioxide, potassium molybdate, and sodium molybdate.

[Claim 12]

The method for producing tantalate particles according to Claim 10 or 11, wherein the sodium compound is sodium carbonate or sodium molybdate, and the potassium compound

is potassium carbonate or potassium molybdate.

[Claim 13]

The method for producing tantalate particles according to any one of Claims 10 to 12, wherein a firing temperature for firing the tantalum compound is 700 to 1500°C.

[Claim 14]

The method for producing tantalate particles according to any one of Claims 10 to 13, the method comprising the steps of:

forming a mixture by mixing together the tantalum compound, the molybdenum compound, and the at least one of the potassium compound and the sodium compound; and

firing the mixture, wherein

a molar ratio of molybdenum atoms to tantalum atoms in the mixture is 0.01 to 5.

FIG. 1

EXAMPLE 1

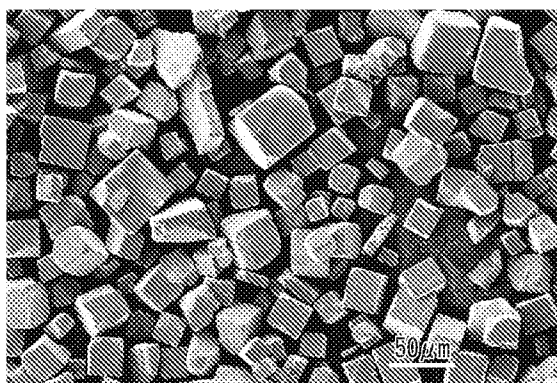


FIG. 2

EXAMPLE 2

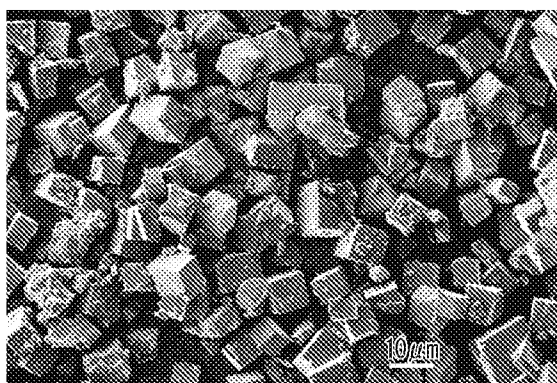


FIG. 3

EXAMPLE 3

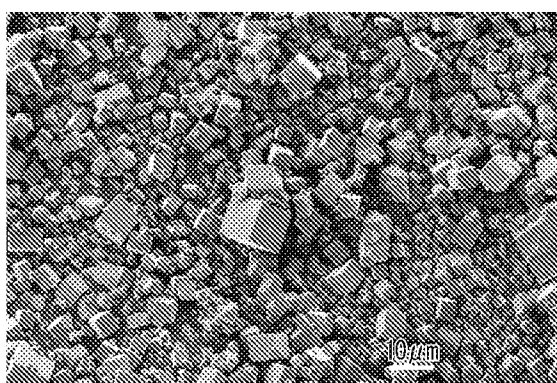


FIG. 4

EXAMPLE 4

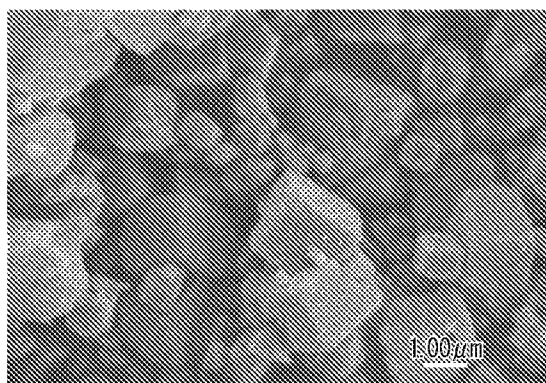


FIG. 5

EXAMPLE 5

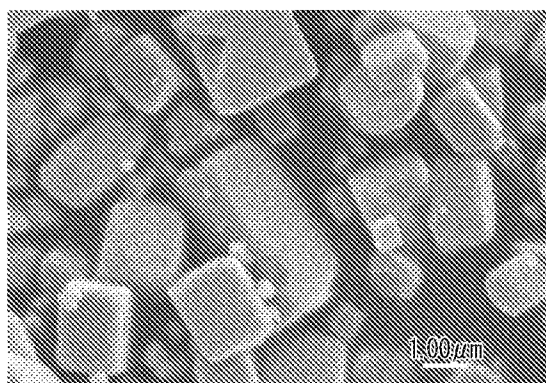


FIG. 6

EXAMPLE 6

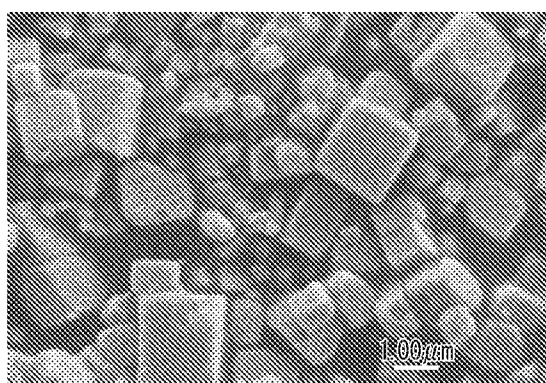


FIG. 7

COMPARATIVE EXAMPLE 1

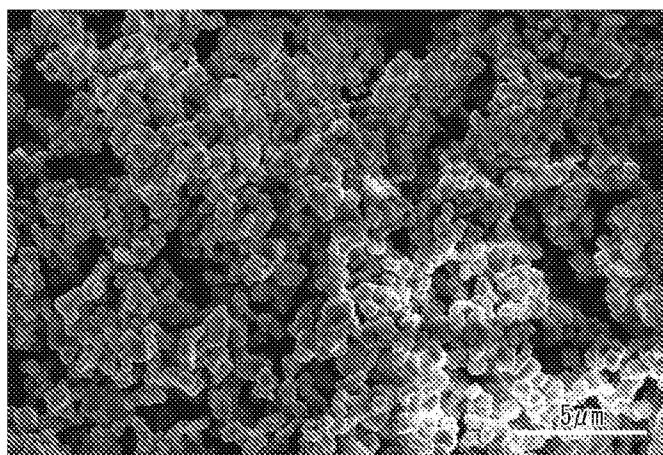


FIG. 8

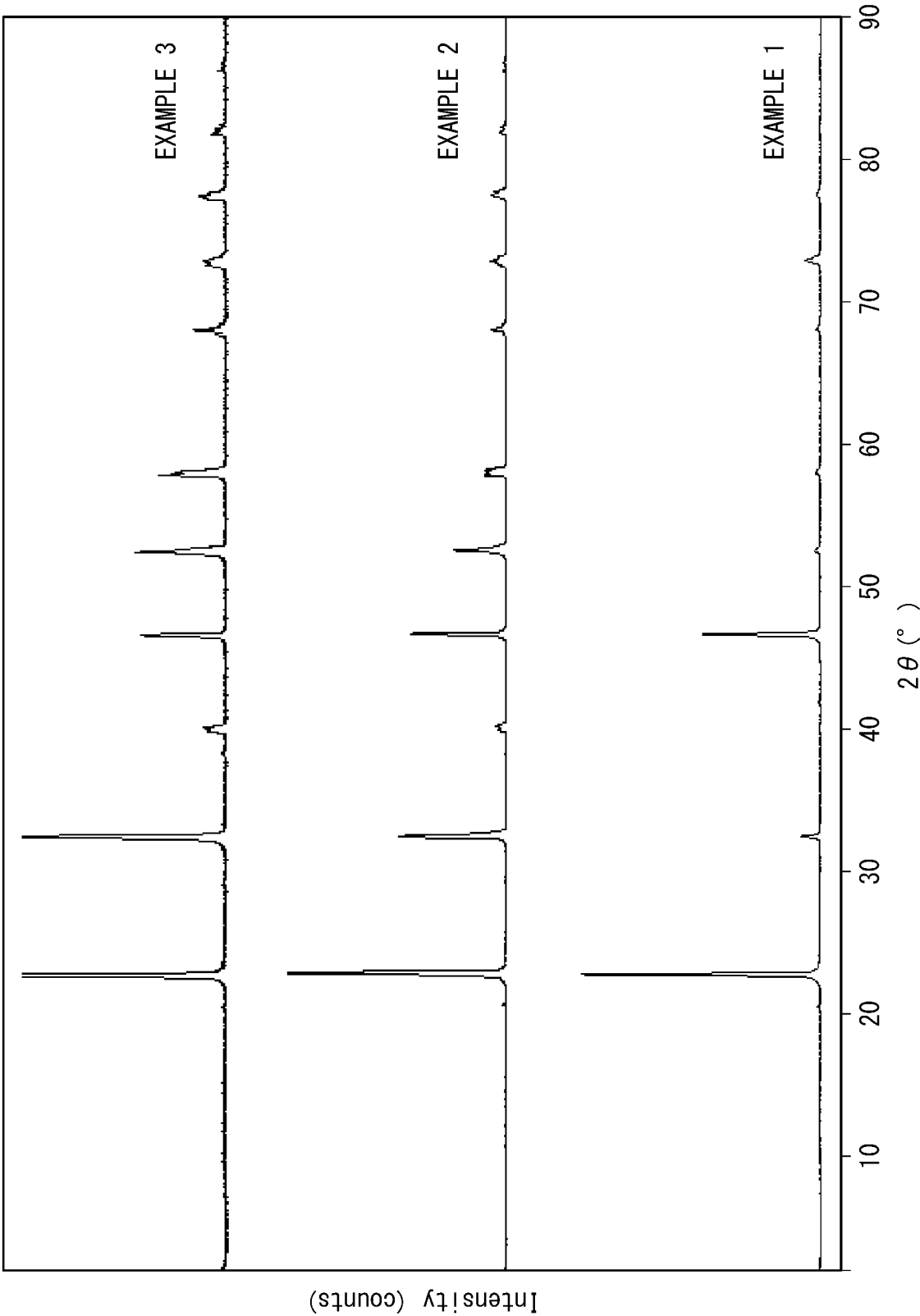


FIG. 9

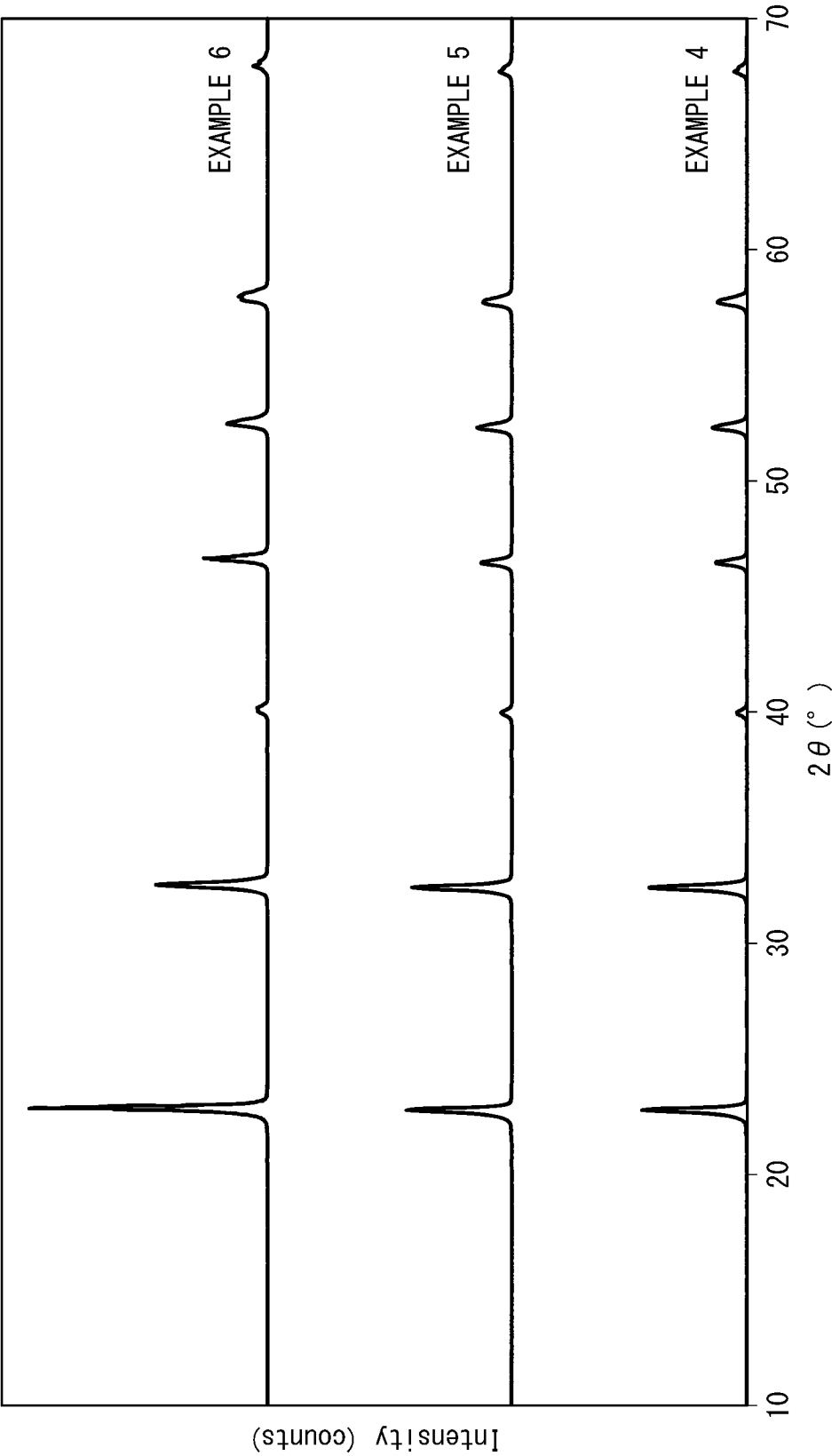


FIG. 10

EXAMPLE 7

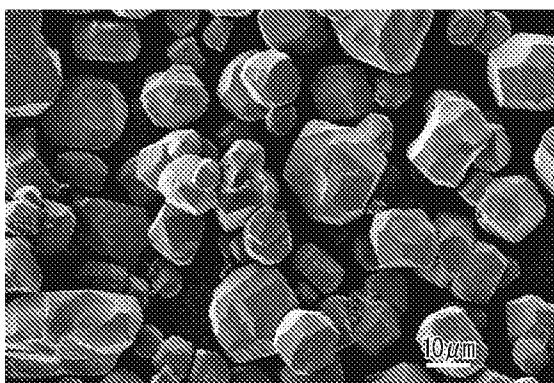


FIG. 11

EXAMPLE 8

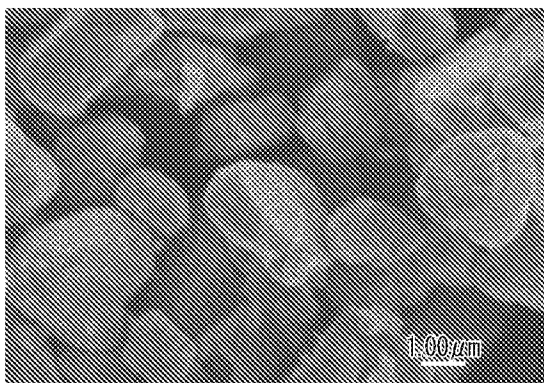


FIG. 12

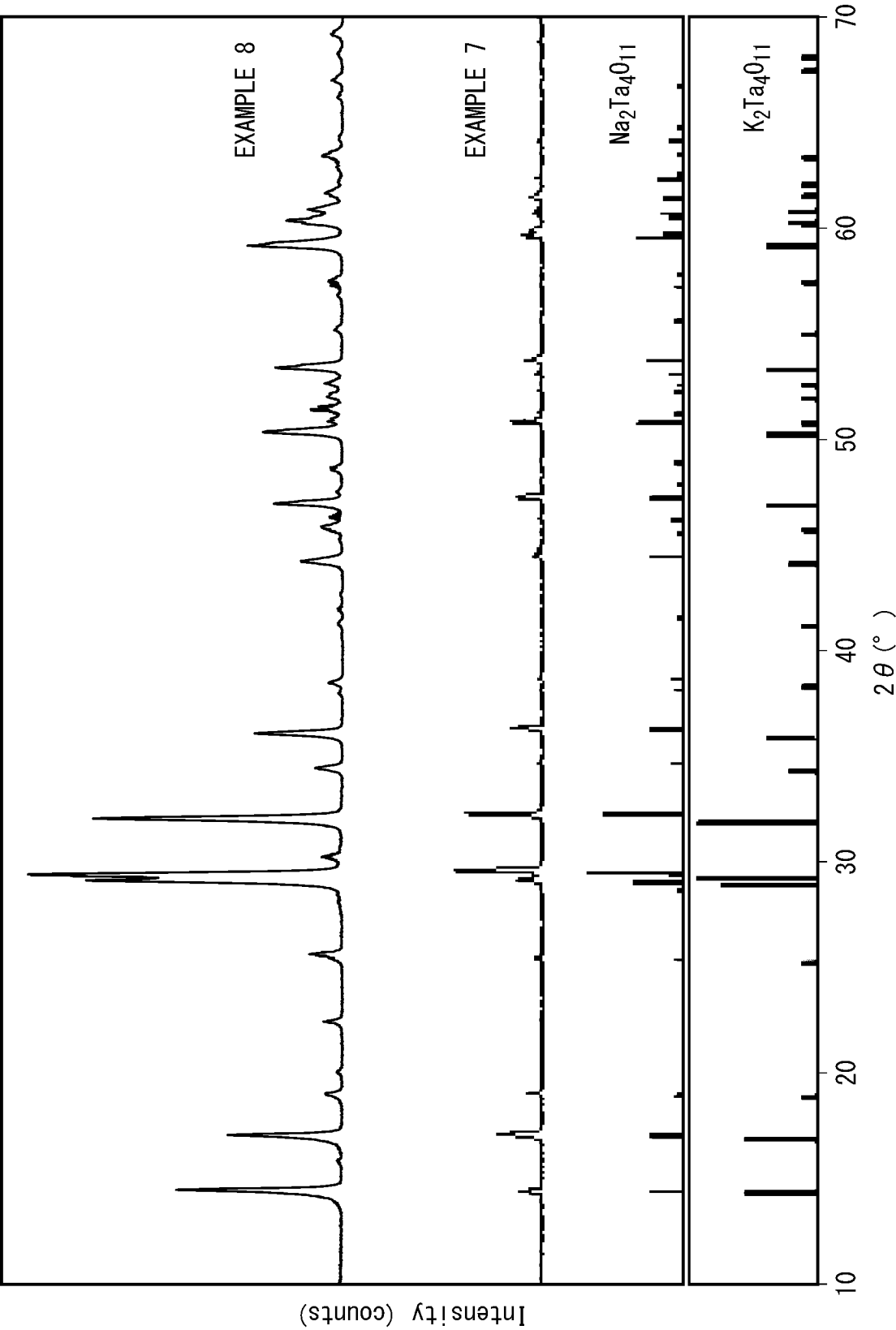


FIG. 13

EXAMPLE 9

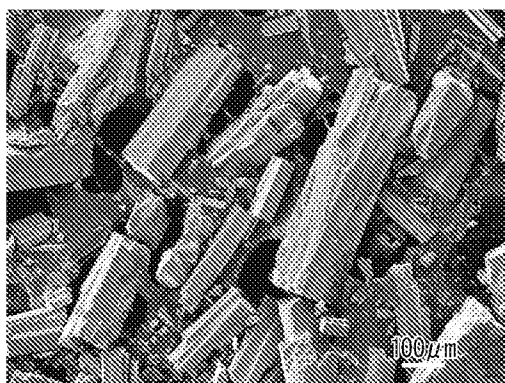


FIG. 14

EXAMPLE 10

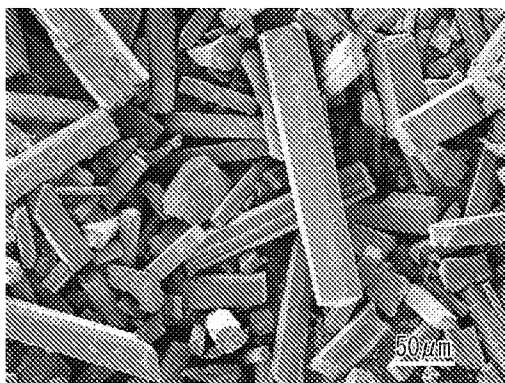


FIG. 15

EXAMPLE 11

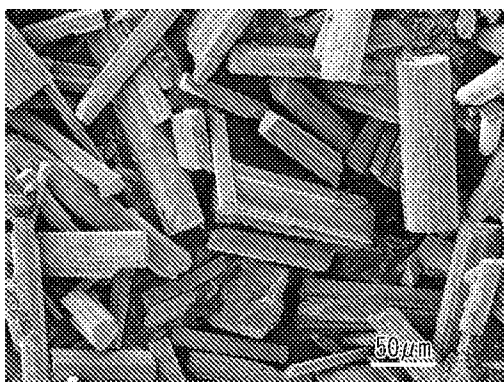


FIG. 16

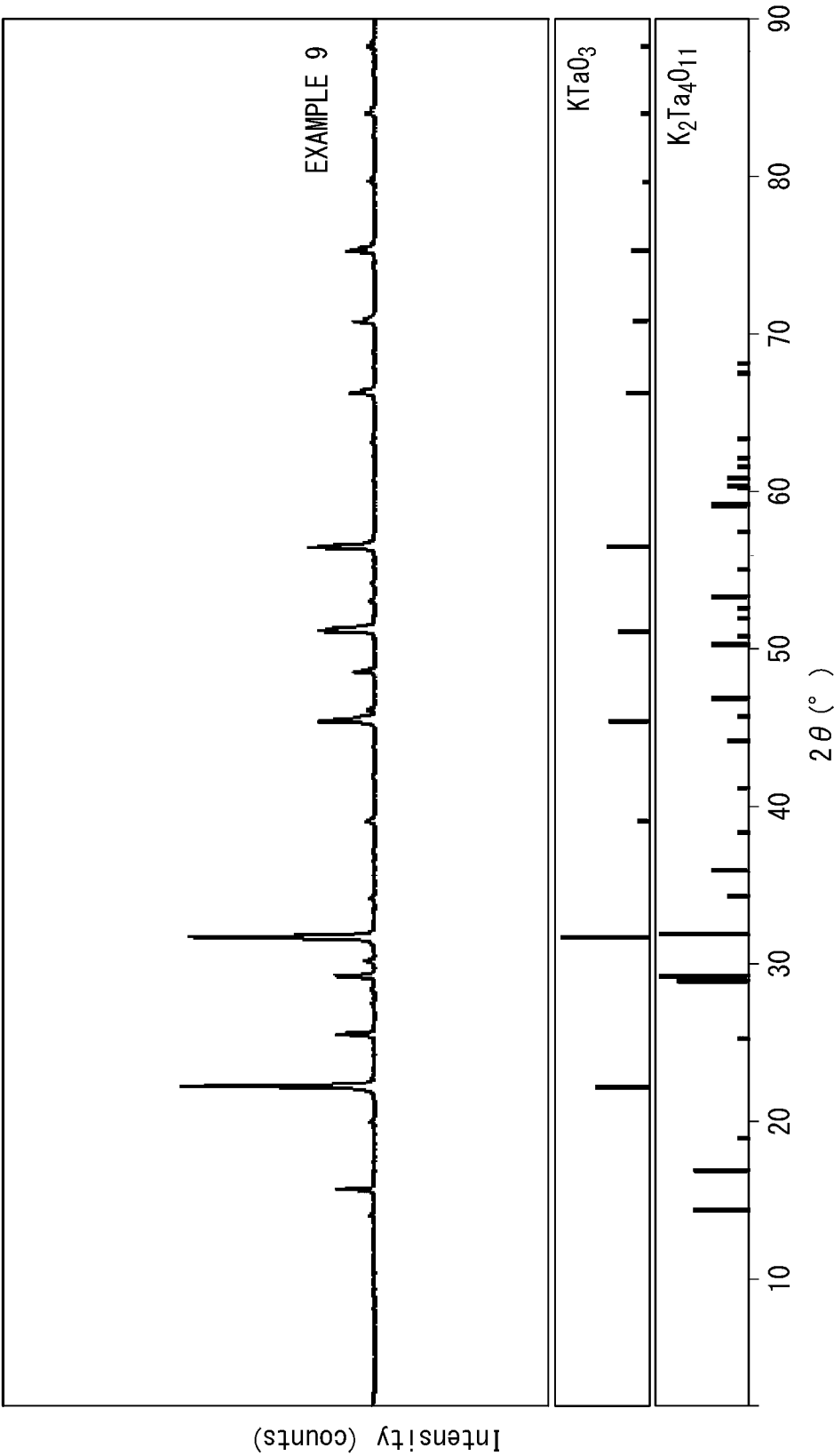
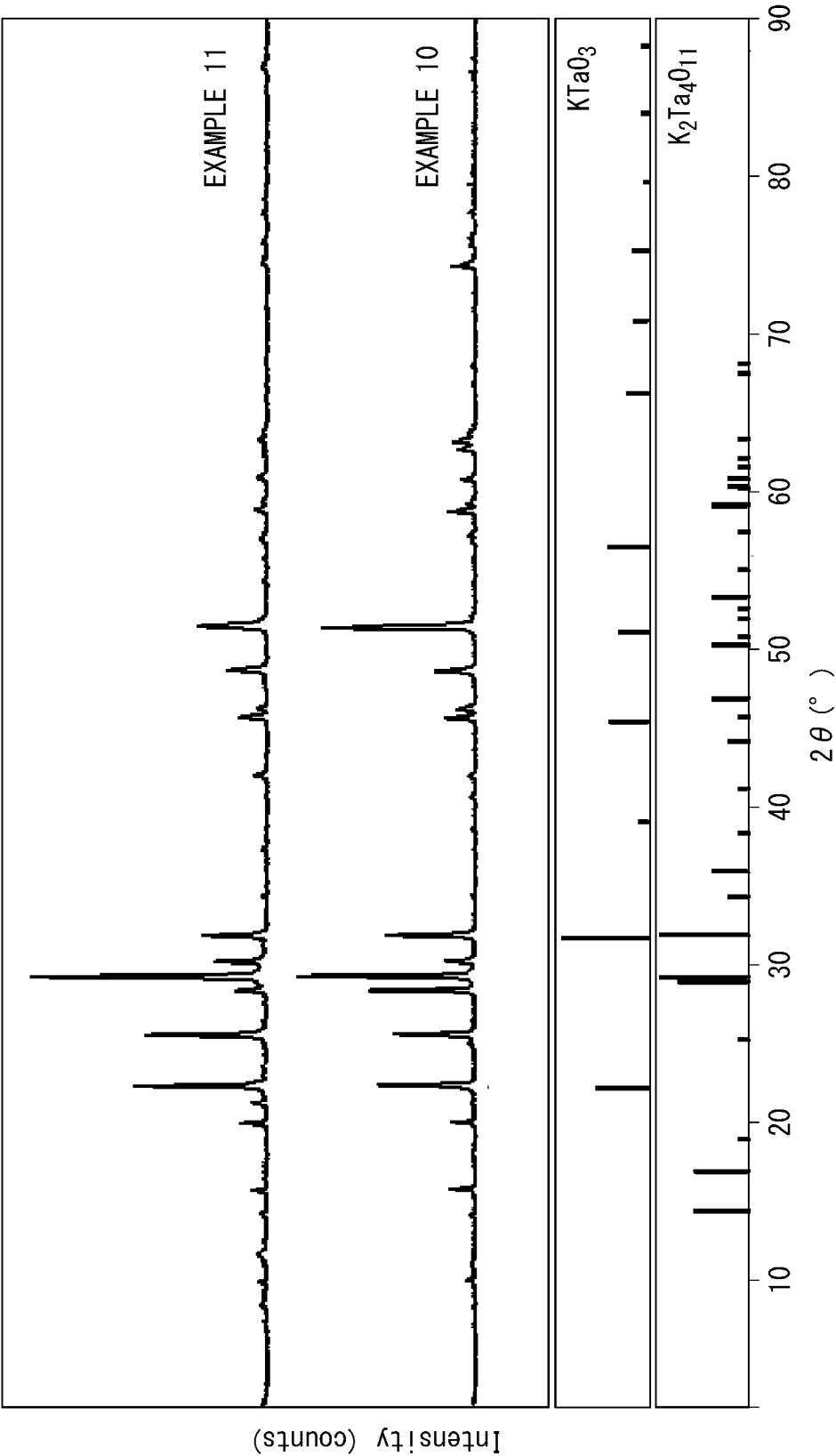


FIG. 17



INTERNATIONAL SEARCH REPORT

International application No.

PCT/CN2022/088141

A. CLASSIFICATION OF SUBJECT MATTER

C01G 35/00(2006.01)i

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

C01G

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

CNABS, CNTXT, CNKI, SIPOABS, DWPI, WOTXT, USTXT, EPTXT, STN, ISI Web of Science, Elsevier Science Direct; DIC CORPORATION; KxNa(1-x)TaO₃, Na₂Ta₄O₁₁, K₂Ta₄O₁₁, KxNa (1-x) TaO₃, natrotantite, molybdenum, Mo, Potassium tantalate, KTaO₃, Sodium tantalate, NaTaO₃, cubic, polyhedral, flux, MoO₃, Na₂MoO₄, K₂MoO₄,

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	TESHIMA, K. et al. "Growth of Na ₂ Ta ₄ O ₁₁ Crystals from a Na ₂ Mo ₂ O ₇ Flux" <i>Crystal Growth & Design</i> , Vol. 6, No. 1, 06 October 2005 (2005-10-06), ISSN: 1528-7505, pp.18-19, page 18, left column last paragraph- right column second paragraph	1-3, 5, 7-14
X	MODAK, B. et al. "Photocatalytic Activity of NaTaO ₃ Doped with N, Mo, and (N, Mo): A Hybrid Density Functional Study" <i>The Journal of Physical Chemistry C</i> , Vol. 118, 01 May 2014 (2014-05-01), ISSN: 1932-7455, pp.10711-10719, page 10714, 3.1.3. Mo-Doped NaTaO ₃	1-4, 7-14
X	BABARY, A. A. et al. "K ₂ Ta ₄ O ₁₁ ("kalitanti") : a wide band gap semiconductor synthesized in molybdate flux medium" <i>CrystEngComm</i> , Vol. 15, 10 May 2013 (2013-05-10), ISSN: 1466-8033, pp. 5539-5544, page 5539, Experimental section	1-3, 6-14



Further documents are listed in the continuation of Box C.



See patent family annex.

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Date of the actual completion of the international search

04 December 2022

Date of mailing of the international search report

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Name and mailing address of the ISA/CN

National Intellectual Property Administration, PRC
6, Xitucheng Rd., Jimen Bridge, Haidian District, Beijing
100088, China

Facsimile No. (86-10)62019451

Authorized officer

YANG, Kun

Telephone No. (86-10)53962711

INTERNATIONAL SEARCH REPORT

International application No.

PCT/CN2022/088141

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	SUZUKI, S. et al. "Growth of Millimeter-sized Platy Single Crystals of NaTaO ₃ from Na ₂ MoO ₄ Flux" 《Crystal Growth & Design》, Vol. 19, 14 June 2019 (2019-06-14), ISSN: 1528-7505, pp.3607–3611, pages 3607 Abstract	1-4, 7-14
A	CN 101439284 A (哈尔滨工业大学) 27 May 2009 (2009-05-27) claims 1-10	1-14
A	CN 1597097 A (南京大学) 23 March 2005 (2005-03-23) claims 1-7	1-14
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