

Description

Title of Invention:

PNEUMATIC SEPARATION APPARATUS FOR
POLYCRYSTALLINE SILICON PULVERIZATION MIXTURES,
5 AND METHOD FOR PRODUCING CHUNK-LIKE MATERIAL,
CHIP-LIKE MATERIAL OR POWDER-LIKE MATERIAL FROM
POLYCRYSTALLINE SILICON PULVERIZATION MIXTURE
USING SAID APPARATUS

10 Technical Field

[0001]

The present invention relates to a crushed
polycrystalline silicon mixture air sorting device. Further,
the present invention relates to a method for manufacturing
15 chunk material, chip material, or powder material from a
crushed polycrystalline silicon mixture with use of the air
sorting device.

Background Art

20 [0002]

The Siemens process is known as a method for
producing polycrystalline silicon also referred to as
polysilicon. The Siemens process involves heating a silicon
core wire placed inside a bell-jar type reactor to silicon

deposition temperature by passing current through the silicon core wire, supplying, to this, gas of a silane compound such as trichlorosilane (SiHCl_3) or monosilane (SiH_4) and hydrogen, and depositing polycrystalline silicon
5 on the silicon core wire by the chemical vapor deposition method, so as to provide a high-purity polycrystalline silicon rod.

[0003]

The polycrystalline silicon rod thus obtained is
10 crushed to a size suitable for a device used in the next step or production of an object to be manufactured in the next step, sorted, and conveyed to the next step. Specifically, the polycrystalline silicon rod is crushed with use of, for example, a hammer made of hard metal such as tungsten
15 carbide to obtain raw material polycrystalline silicon blocks. Then, the raw material polycrystalline silicon blocks are further crushed to a desired particle size with use of, for example, a crushing device made of rigid polymer or hard metal or the aforementioned hammer, and a crushed
20 polycrystalline silicon mixture thus obtained is then classified according to need into a desired size with use of a classification device made of the same material to obtain a crushed polycrystalline silicon material having a desired particle size.

[0004]

The crushed polycrystalline silicon material is referred to as, for example, "dust", "powder material", "chip material", and "chunk material" in accordance with its size. In the
5 present specification, the material having a particle size of less than 500 μm is referred to as "dust", the material having a particle size of not less than 500 μm and less than 1000 μm is referred to as "powder material", the material having a particle size of not less than 1000 μm to less than 1500
10 μm is referred to as "chip material", and the material having a particle size of not less than 1500 μm is referred to as "chunk material". These are favorably used selectively in accordance with the filling property to a crucible and the solubility according to their particle sizes, as raw materials
15 for manufacture of monocrystalline silicon, which are raw materials for semiconductor wafers or solar power generation wafers.

[0005]

The crushed polycrystalline silicon mixture containing
20 the crushed polycrystalline silicon materials differing in particle size is typically classified with use of a mechanical sieve screening machine. For example, a vibration sieve screening machine performing sieving by vibration and a sieve screening machine performing sieving by rotation of a

rotation cylinder are known. However, with such a mechanical sieve screening machine, the crushed polycrystalline silicon material inevitably experiences severe surface metal contamination because of excessive contact
5 with metal material of the sieve screening part. This can be somewhat mitigated by lining, with resin, a contact portion with the metal material of the sieve screening part. However, crushed surfaces of the crushed polycrystalline silicon material include acute corner portions and protruding
10 portions, and thus vibration of the crushed material for a long time period or with intensity has resulted in unacceptable metal contamination after all due to, for example, scraping of the resin lining. Further, it has been concerned that the crushed polycrystalline silicon material
15 experiences carbon contamination due to the resin pieces scraped off.

[0006]

The crushed polycrystalline silicon material is mainly used as raw materials of semiconductor or solar power
20 generation wafers. In this case, reduction in metal impurities is one of the strongest quality requirements. Therefore, in order to prevent contact between the crushed polycrystalline silicon material and metal material as much as possible, it has been proposed that the above classification is performed

by air (see, for example, [Fig. 1] of Patent Literature 1 and
paragraph [0064] of Patent Literature 2). That is, Patent
Literature 1 discloses causing semiconductor fragments
such as polycrystalline silicon to fall by gravity downwards,
5 applying an air flow during the falling, from a nozzle with its
jet orifice facing horizontally, horizontally blowing away ones
with small particle sizes contained in the semiconductor
fragments, and classifying the semiconductor fragments by
the particle sizes in accordance with the travel distances.
10 Further, Patent Literature 2 discloses remove dust with a
small particle diameter by sucking by, while causing the
crushed polycrystalline silicon material to fall by gravity
similarly, sucking it horizontally, unlike Patent Literature 1
above.

15

Citation List

[Patent Literature]

[0007]

[Patent Literature 1]

20 Japanese Patent Application Publication *Tokukai* No.
2000-070859

[Patent Literature 2]

Japanese Patent Application Publication *Tokukai* No.
2016-079053

Summary of Invention

Technical Problem

[0008]

5 However, such a conventional method by air classification provides insufficient sorting effect. Therefore, unfortunately, in particular, the chunk material and the chip material that have large particle sizes and are heavy among the crushed polycrystalline silicon mixture cannot be
10 classified with high accuracy. For example, in above Patent Literature 2 in which the crushed polycrystalline silicon mixture is horizontally sucked during the falling by gravity, materials up to dust and powder material can be removed, and the chip material and the chunk material are to be
15 sorted in the next step with use of a sieve screening machine or a classification roller.

[0009]

 In view of such a background, it is an object of the present invention to, with respect to the air classification
20 device (air sorting device) excellent in reduction in surface metal contamination of crushed polycrystalline silicon material obtained, develop an air classification device that can sophisticatedly classify even the chunk material and the

chip material that are heavy by further improving the sorting effect thereof.

Solution to Problem

[0010]

5 In light of the aforementioned object, the present inventors have performed diligent study. As a result, the present inventors have found that the object is attained by achieving a device having a specific structure of applying a suction force from above to a crushed polycrystalline silicon
10 mixture falling by gravity, and have made the present invention.

[0011]

That is, the present invention provides a crushed polycrystalline silicon mixture air sorting device including:
15 a first air sorting tube part that includes a tubular body provided so as to stand vertically and that is provided with an inlet for a crushed polycrystalline silicon mixture containing chunk material and small particle diameter material having a smaller particle diameter than that of the
20 chunk material, the inlet being formed in an axially middle area of the tubular body, the first air sorting tube part being configured to perform air sorting between the chunk material and the small particle diameter material, wherein

the chunk material in the crushed polycrystalline silicon mixture that has been introduced into the tubular body falls downwards, and a flow tube path for the small particle diameter material is connected to an upper end of
5 the first air sorting tube part and is provided with a sucking section located upstream of the flow tube path, the sucking section being configured to apply a suction force for sucking the small particle diameter material upwards.

10 [0012]

Further, the present invention also provides a crushed polycrystalline silicon mixture air sorting device according to the above crushed polycrystalline silicon mixture air sorting device, in which a second air sorting tube part that
15 includes a tubular body provided so as to stand vertically midway in the flow tube path and that is provided with an inflow port for the small particle diameter material from the flow tube path is interposed, the inflow port being formed in an axially middle area of the tubular body,

20 the tubular body having a diameter adjusted so as to apply a suction force that allows chip material in the small particle diameter material that has entered the tubular body to fall downwards and that sucks powder material having a smaller particle diameter than that of the chip material

upwards, the second air sorting tube part being configured to perform air sorting between the chip material and the powder material.

5 [0013]

Further, the present invention also provides a method for manufacturing chunk material from a crushed polycrystalline silicon mixture, the method including: introducing, with use of the crushed polycrystalline silicon
10 mixture air sorting device according to claim 1, a crushed polycrystalline silicon mixture into the tubular body of the first air sorting tube part; sorting the crushed polycrystalline silicon mixture by air; and collecting the chunk material from a lower end of the tubular body.

15 [0014]

Further, the present invention also provides a method for manufacturing chip material from a crushed polycrystalline silicon mixture, the method including: using the above crushed polycrystalline silicon mixture air sorting
20 device to sort, by air, the small particle diameter material that has entered the tubular body of the second air sorting tube part; and collecting the chip material from a lower end of the tubular body or similarly,

a method for manufacturing powder material from a crushed polycrystalline silicon mixture, the method including: using the above crushed polycrystalline silicon mixture air sorting device to sort, by air, the small particle
5 diameter material that has entered the tubular body of the second air sorting tube part; and collecting the powder material that has exited from an upper end of the tubular body to the flow tube path.

Advantageous Effects of Invention

10 [0015]

According to the present invention, it is possible to, with respect to an air sorting device excellent in reduction in surface metal contamination of crushed polycrystalline silicon material obtained, further improve a sorting effect
15 thereof and sophisticatedly sort even chunk material and chip material that are heavy.

Brief Description of Drawings

[0016]

20 Fig. 1 is a cross-sectional view schematically illustrating a crushed polycrystalline silicon mixture air sorting device in accordance with an embodiment of the present invention.

Description of Embodiments

[0017]

The crushed polycrystalline silicon mixture classified by a device in accordance with the present invention
5 contains at least one selected from, for example, a chunk material and a small particle diameter material having a smaller particle diameter than that of the chunk material, i.e., a powder material and a chip material. Such a crushed polycrystalline silicon mixture may be derived from any
10 material without limitation, but is typically obtained by crushing raw material polycrystalline silicon blocks. The raw material polycrystalline silicon blocks are typically obtained by crushing a polycrystalline silicon rod obtained by the Siemens process, with use of, for example, a hammer made
15 of hard metal such as tungsten carbide.

[0018]

Such a crushed polycrystalline silicon mixture preferably contains the chunk material having a particle size of not less than 1500 μm in an amount of 80% by mass to
20 98.5% by mass, in particular, 85% by mass to 98% by mass.

[0019]

Further, it is suitable that the crushed polycrystalline silicon mixture contains the small particle diameter material having a smaller particle diameter than that of the chunk

material in an amount of 1.5% by mass to 20% by mass, which accounts for the rest other than the preferable amount of the chunk material contained. Among this, the chip material having a particle size of not less than 1000 μm to
5 less than 1500 μm is preferably contained in an amount of 0.5% by mass to 10% by mass, in particular, 1% by mass to 9% by mass relative to the crushed polycrystalline silicon mixture. Further, the powder material having a particle size of not less than 500 μm and less than 1000 μm is preferably
10 contained in an amount of 0.5% by mass to 10% by mass, in particular, 1% by mass to 9% by mass relative to the crushed polycrystalline silicon mixture.

[0020]

Note that, as described above, the crushed
15 polycrystalline silicon mixture contains the dust having a particle size (less than 500 μm) further smaller than that of the powder material (typically in an amount of not more than 1% by mass relative to the crushed polycrystalline silicon mixture) in addition to the chunk material, the chip material,
20 and the powder material. The dust composed of such extremely small particles tends to experience more severe surface metal contamination than other ones in form having larger particle sizes, and is typically useless. The dust is relatively easy to remove from the crushed polycrystalline

silicon mixture because of its lightness, and thus, as described in Patent Literature 2, can be favorably removed even by horizontally applying a moderate suction force during the falling by gravity in the crushing device after
5 crushing. Further, the dust can be effectively removed while being conveyed on a conveyor after the crushing even by applying a moderate suction force from above the placed crushed polycrystalline silicon mixture. Therefore, one suitable aspect is such that most of the dust is removed from
10 the crushed polycrystalline silicon mixture with use of these means before the crushed polycrystalline silicon mixture is supplied to an air sorting device in accordance with the present invention.

[0021]

15 The crushed polycrystalline silicon mixture preferably has a surface metal concentration of 0.3 ppbw to 10 ppbw, in particular, 0.4 ppbw to 9 ppbw. Note that in the present invention, the surface metal concentration of crushed polycrystalline silicon material refers to a value measured by
20 the following method. That is, it is a method for analytical determination, by inductively coupled plasma-mass spectrometry (ICP-MS), of each metal element content in a sample liquid obtained by decomposing and removing a surface of crushed polycrystalline silicon material to be

measured. Specifically, into a clean polytetrafluoroethylene beaker having a capacity of 500 ml, approximately 400 g of crushed polycrystalline silicon material was transferred. Then, 100 ml of a solution (50% by mass - HF: 10 ml and 5 70% by mass - nitric acid: 90 ml) was added, and extraction was performed at 25°C for 15 minutes. Liquid contents in the polytetrafluoroethylene beaker and cleaning water that was obtained by cleaning a surface of the crushed polycrystalline silicon material with use of 100 ml of 10 ultrapure water were transferred into a clean polytetrafluoroethylene beaker, and were regarded as an extraction solution from a surface (surface extraction solution) of the crushed polycrystalline silicon material. The surface extraction solution of the crushed polycrystalline 15 silicon material was evaporated to dryness, and a 3.5% by mass - nitric acid aqueous solution was added to the surface extraction solution evaporated, so that a volume was adjusted to 20.0 ml. ICP-MS measurement was then performed to calculate metal concentrations of Na, Mg, Al, K, 20 Ca, Cr, Fe, Ni, Co, Cu, Zn, W, Ti, and Mo, which were calculated as a concentration value relative to the total mass of the crushed polycrystalline silicon mixture.

[0022]

An air sorting device in accordance with the present invention is a device that accurately classifies the crushed polycrystalline silicon mixture described above into a chunk material having a large particle size and a small particle diameter material having a smaller particle diameter than it. It is characterized by the feature of including: a first air sorting tube part that includes a tubular body provided so as to stand vertically and that is provided with an inlet for the crushed polycrystalline silicon mixture, the inlet being formed in an axially middle area of the tubular body, the first air sorting tube part being configured to perform air sorting between the chunk material and the small particle diameter material, wherein

the chunk material in the crushed polycrystalline silicon mixture that has been introduced into the tubular body falls downwards, and a flow tube path for the small particle diameter material which path is connected to an upper end of the first air sorting tube part and is provided with a sucking section located upstream of the flow tube path, the sucking section being configured to apply a suction force for sucking the small particle diameter material upwards.

[0023]

The details thereof will be described with reference to Fig. 1, which is a cross-sectional view schematically illustrating a representative aspect. In Fig. 1, an air sorting device 1 includes a first air sorting tube part 2 made up of a tubular body provided so as to stand vertically and configured to sort between the chunk material and the small particle diameter material. As this first air sorting tube part 2, one having a straight tube shape with the same diameter is typically used, but one provided partially with part having a different diameter or tapered one having a diameter becoming narrower in an upward direction or downward direction may be used. The diameter of the tubular body is not specifically determined because being affected by a particle diameter distribution and an introduction amount of crushed polycrystalline silicon mixture introduced, but the diameter is preferably large enough to allow an opening area of the tubular body (area of an opening of the tubular body made in a case where the tubular body is cut in a plane perpendicular to the axial direction of the tubular body) to be 25 cm² to 2500 cm². More preferably, the diameter of the tubular body is large enough to allow the opening area of the tubular body to be 100 cm² to 1600 cm². Further, its axial length is preferably 50 cm to 200 cm, more preferably 70 cm to 180 cm.

[0024]

The first air sorting tube part 2 is provided with an inlet 3 for the crushed polycrystalline silicon mixture which is formed in an axially middle area thereof. Here, the axially middle area of the first air sorting tube part 2 is defined as an area of $1/4$ to $3/4$, preferably $1/3$ to $2/3$ of the total axial length of the air sorting tube part upward from its lower end. In other words, the axially middle area of the first air sorting tube part 2 is defined as an area of not less than $1/4$ and not more than $3/4$, preferably, not less than $1/3$ and not more than $2/3$ from the lower end of the first air sorting tube part 2 with respect to a vertical length of the first air sorting tube part 2.

[0025]

A crushed polycrystalline silicon mixture 4 prepared by crushing using, for example, a raw material polycrystalline silicon block crushing device or a hammer is conveyed by a feeder 5 into the inlet 3 for the crushed polycrystalline silicon mixture of the first air sorting tube part 2 and is introduced into the first air sorting tube part 2 therethrough. It is introduced at preferably, though not limited to, by an amount of 10 kg/minute to 150 kg/minute, more preferably, 15 kg/minute to 100 kg/minute. Further, the crushed polycrystalline silicon mixture 4 is conveyed by

the feeder 5 preferably at a speed of 5 m/minute to 20 m/minute, more preferably 7 m/minute to 18 m/minute immediately before introduction into the first air sorting tube part 2. The crushed polycrystalline silicon mixture 4 is
5 preferably introduced in a state in which each crushed material is uniformly dispersed and do not overlap as much as possible on the feeder 5.

[0026]

The gravity acts on the crushed materials of which the
10 crushed polycrystalline silicon mixture 4 that has been introduced into the first air sorting tube part 2 as described above is composed, the gravity causing the crushed materials to fall downwards in the tubular body. In the present invention, in the tubular body of the first air sorting
15 tube part 2, such gravity lets the chunk material having a large particle diameter fall downwards, whereas to the small particle diameter material, a suction force strong enough to suck the small particle diameter material upwards in the tubular body against the gravity is applied from above.
20 Therefore, the crushed polycrystalline silicon mixture 4 that has been introduced into the tubular body is sorted into the chunk material 6 and the small particle diameter material 7. The chunk material 6 that in turn falls downwards is taken out of a vent 8 for the chunk material at the lower end of the

first air sorting tube part 2 and is collected. The small particle diameter material 7 that is in turn sucked upwards enters the flow tube path 9 for the small particle diameter material which is connected to the upper end opening of the
5 sorting tube part, and flows upstream.

[0027]

A sucking section for applying, to the inside of the tubular body of the first air sorting tube part 2, a suction force suitably strong for sorting between the chunk material
10 6 and the small particle diameter material 7 is connected upstream of the flow tube path 9 for the small particle diameter material. As the sucking section, for example, a dust collector or a sucking pump can be used without any limitation, but a collecting section for the small particle
15 diameter material is preferably provided midway in the flow tube path 9 for the small particle diameter material up to its sucking opening. Although described later in detail, a second air sorting tube part 11 is preferably interposed midway in the flow tube path 9 for the small particle diameter material,
20 the second air sorting tube part 11 being configured to sort, by air, the small particle diameter material 7 flowing here into the chip material and the powder material. In this case, as the sucking section, a dust collector 10 is preferably used. The dust collector 10 generates a suction force by, for

example, rotating a fan by a motor, and fine material contained in the sucked air can be collected by a filter 19. Therefore, through the second air sorting tube part 11, the powder material flows, together with dust, upstream of the
5 flow tube path 9 for the small particle diameter material. They are sucked by the dust collector 10 and then are collected by the filter 19.

[0028]

The suction force applied to the inside of the tubular
10 body of the first air sorting tube part 2 by the sucking section such as the dust collector 10 is not specifically determined because being affected by a particle diameter distribution and an introduction amount of the crushed polycrystalline silicon mixture to be sorted. However,
15 typically, the chunk material 6 and the small particle diameter material 7 are favorably sorted between in a case where an upward wind speed is not less than 7 m/s and not more than 20 m/s, more suitably, not less than 8 m/s and not more than 17 m/s, and thus the suction force may be set
20 in accordance with the diameter of the tubular body so as to provide part with such a wind speed in the first air sorting tube part 2. Here, the wind speed is calculated as:

Wind speed $V = \text{air volume } Q / \text{area } S \text{ passed}$
(cross-sectional area of tubular body)

Note that the area S refers to the opening area of the tubular body.

[0029]

In order to more accurately sort between the chunk
5 material 6 and the small particle diameter material 7, it is
preferable to set the suction force so as to achieve the above
wind power in a range from 10 cm below a lower end of the
opening of the inlet 3 for the crushed polycrystalline silicon
mixture, more suitably, from 15 cm below the lower end to
10 10 cm above an upper end of the opening of the inlet, more
suitably, to 15 cm above the upper end in the tubular body
of the first air sorting tube part 2.

[0030]

The air sorting device 1 in Fig. 1 is provided with a
15 second air sorting tube part 11 interposed midway up to an
upstream area in which the sucking section is provided in
the flow tube path 9 for the small particle diameter material
that is connected to the upper end of the first air sorting
tube part 2. The second air sorting tube part 11 is configured
20 to sort the small particle diameter material 7 flowing here
after the sorting by the first air sorting tube part 2, into the
chip material and the powder material. As with the first air
sorting tube part 2, the second air sorting tube part 11 has
a tubular body provided so as to stand vertically and is

provided with an inflow port 12 for the small particle diameter material from the flow tube path 9 for the small particle diameter material, the inlet being formed in an axially middle area of the tubular body. The definition of this axially middle area is the same as that specified for the first air sorting tube part. The tubular body of the second air sorting tube part 11 is provided so as to stand vertically midway in the flow tube path 9.

[0031]

10 The second air sorting tube part 11 is located midway in the flow tube path 9 for the small particle diameter material and provided with the sucking section such as the dust collector 10 located upstream. A suction force is thus applied to the inside of the tubular body from above.

15 Therefore, one important feature of the present invention is such that the diameter of the tubular body is adjusted so as to make the suction force strong enough to, among the small particle diameter material 7 that has entered the tubular body, let the chip material fall downwards and suck the

20 powder material having a smaller particle diameter than that of the chip material upwards. Therefore, the small particle diameter material 7 that has entered the tubular body is sorted into the chip material 13 and the powder material 14. The chip material 13 (and, if any, remaining chunk material

that has not sorted by the first air sorting tube part 2 and is left in the flow tube path 9 for the small particle diameter material) that in turn falls downwards is taken out of a vent 15 for the chip material at a lower end of the second air sorting tube part 11 and is collected. Meanwhile, the powder material 14 (and dust) that is in turn sucked upwards enters the flow tube path 9 for the small particle diameter material again from the upper end opening of the sorting tube part and flows further upstream.

10 [0032]

The adjustment of the diameter of the tubular body of the second air sorting tube part 11 is not be specifically determined because being affected by, for example, a particle diameter distribution, an inflow amount of the small particle diameter material 7 to be sorted, and the length of the tube path of the flow tube path 9 for the small particle diameter material. However, the chip material 13 and the powder material 14 are favorably sorted between when an upward wind speed is not less than 2 m/s and less than 7 m/s, more preferably, not less than 3 m/s and not more than 6 m/s, and thus the diameter of the tubular body may be set so as to provide part with such a wind speed in the tubular body of the first air sorting tube part 2. In order to accurately sort between the chip material 13 and the powder material 14, it

is preferable to set the suction force so as to achieve the
above wind power in a range from 10 cm below a lower end
of the opening of the inflow port 12 for the small particle
diameter material, more suitably, from 15 cm below the lower
5 end to 10 cm above an upper end of the opening of the inlet,
more suitably, to 15 cm above the upper end in the tubular
body of the second air sorting tube part 11.

[0033]

Therefore, the diameter of the tubular body of the
10 second air sorting tube part 11 may be adjusted to satisfy
this sucking condition. To satisfy the condition, the tubular
body of the second air sorting tube part 11 needs to have a
larger diameter than that of the tubular body of the first air
sorting tube part 2. Specifically, as the diameter of the
15 tubular body of the second air sorting tube part 11, a size
that satisfies the condition is preferably selected from a
range that allows the opening area of the tubular body of the
second air sorting tube part 11 to be approximately 3 times
to 7 times, in particular, approximately 3.5 times to 6.5
20 times larger than the opening area of the tubular body of the
first air sorting tube part 2. The diameter of the tubular body
of the second air sorting tube part 11 is preferably large
enough to allow the opening area of the tubular body of the
second air sorting tube part 11 to be 75 cm² to 17,500 cm²,

more preferably, large enough to allow the opening area of the tubular body of the second air sorting tube part 11 to be 300 cm² to 11,200 cm².

[0034]

5 As this second air sorting tube part 11, one having a straight tube shape with the same diameter is also typically used, but one provided partially with part having a different diameter or tapered one having a diameter becoming narrower in an upward direction or downward direction may
10 be used. The axial length thereof is preferably selected from 50 cm to 250 cm, and is more preferably selected from 60 cm to 200 cm.

[0035]

 In the first half of the flow tube path 9 for the small
15 particle diameter material that connects the second air sorting tube part 11 and the first air sorting tube part 2, the wind speed is preferably set to not less than 7 m/s and not more than 20 m/s, more suitably, not less than 8 m/s and not more than 17 m/s in order to prevent the flowing small
20 particle diameter material from being stuck midway, as with the sorting part for the chunk material 6 and the small particle diameter material 7 in the first air sorting tube part 2. That is, it is favorable to adjust the diameter of the tubular body so as to achieve this wind speed.

[0036]

In the flow tube path 9 for the small particle diameter material, the dust collector 10 is, as described above, provided in the second half located further upstream than
5 the second air sorting tube part 11 interposed. Therefore, the powder material that has entered the tube path after the sorting by the second air sorting tube part 11 is collected by the dust collector 10 and is taken out of a powder material vent 17 thereof. An exhaust port 18 is formed in an upper
10 portion of the dust collector 10, and air separated from the powder material by the filter 19 exits from the exhaust port 18.

[0037]

Note that, as described above, in addition to the chunk
15 material, the chip material, and the powder material, the dust is typically contained in a slight amount in the crushed polycrystalline silicon mixture 4 produced by crushing using, for example, a polycrystalline silicon block crushing device or a hammer. In a case where such dust is supplied to the
20 air sorting device 1 without being removed in advance, the dust is sorted together with the powder material because of its small particle size, and exits together with the powder material from the powder material vent 17. The powder material obtained with the dust contained as described

above can be used as raw materials for semiconductor wafers and solar power generation wafers, but is preferably further subjected to mild sucking process to remove the dust mixed therewith as much as possible.

5 [0038]

In the air sorting device described above, the material of the device is not particularly limited, but specifically, metals such as iron and nickel; alloys such as stainless steel and Ni-based alloy (e.g., hastelloy or inconel) are preferable
10 for part where structural strength is need. One favorable aspect is to line the inner surface thereof with, for example, resin in order to reduce surface metal contamination of the crushed polycrystalline silicon material. Further, the part where structural strength is not needed is more preferably
15 made of resin, such as polyvinyl chloride, polyvinylidene fluoride, polypropylene, and polyether ether ketone.

[0039]

The sorting using such an air sorting device enables the crushed polycrystalline silicon mixture to be accurately
20 sorted into the chunk material, the chip material, and the powder material. Such air sorting enables reduction in the contact with metal material, thereby making it possible to manufacture the crushed materials having respective particle sizes that are sorted, with reduced surface metal

contamination. Therefore, the crushed materials thus obtained can be favorably used for the raw materials for semiconductor wafers and solar power generation wafers.

Examples

5 [0040]

The following will describe the present invention on the basis of more detailed Examples, but the present invention is not limited to Examples below. In Examples and Comparative Examples, the physical properties were
10 measured by the following method.

[0041]

[Contents of chunk material, chip material, powder material, and dust contained in crushed polycrystalline silicon mixture]

15 A crushed polycrystalline silicon mixture was sieved with use of each of sieves having sieve openings of 500 μm , 1,000 μm , and 1,500 μm in decreasing order of the sieve openings of the sieves. Then, weights of the materials remaining on the sieves and the final material having passed
20 through the sieves were measured with use of an electronic scale, and each weight ratio was calculated. The weight ratio of the material remaining on the sieve having the sieve opening of 1,500 μm was calculated as a chunk material content, the weight ratio of the sieve having the sieve

opening of 1,000 μm was calculated as a chip material content, the weight ratio of the material remaining on the sieve having the sieve opening of 500 μm was calculated as a powder material content, and the weight ratio of the material having passed through the sieve having the sieve opening of 500 μm was calculated as a dust content.

[0042]

[Surface metal concentration of crushed polycrystalline silicon mixture]

10 Into a clean polytetrafluoroethylene beaker having a capacity of 500 ml, approximately 400 g of crushed polycrystalline silicon material was transferred. Then, 100 ml of a solution (50% by mass - HF: 10 ml and 70% by mass - nitric acid: 90ml) was added, and extraction was performed
15 at 25°C for 15 minutes. Liquid contents in the polytetrafluoroethylene beaker and cleaning water that was obtained by cleaning a surface of the crushed polycrystalline silicon material with use of 100 ml of ultrapure water were transferred into a clean polytetrafluoroethylene beaker, and
20 were regarded as an extraction solution from a surface (surface extraction solution) of the crushed polycrystalline silicon material. The surface extraction solution of the crushed polycrystalline silicon material was evaporated to dryness, and a 3.5% by mass - nitric acid aqueous solution

was added to the surface extraction solution evaporated, so that a volume was adjusted to 20.0 ml. ICP-MS measurement was then performed to calculate metal concentrations of Na, Mg, Al, K, Ca, Cr, Fe, Ni, Co, Cu, Zn, W, Ti, and Mo, which
5 were calculated as a concentration value relative to the total mass of the crushed polycrystalline silicon mixture.

[0043]

Example 1

The crushed polycrystalline silicon mixture 4 was
10 sorted by particle size with use of the air sorting device 1 illustrated in Fig. 1. The main component of which the air sorting device 1 is composed was stainless steel lined with resin because structural strength is need, and part where structural strength is not needed is made of polypropylene
15 or polyvinyl chloride.

[0044]

The diameter of the first air sorting tube part 2 was large enough to allow the opening area of the tubular body of the first air sorting tube part 2 to be 450 cm^2 , and the
20 axial length of the first air sorting tube part 2 was 100 cm. The diameter of the second air sorting tube part 11 was large enough to allow the opening area of the tubular body of the second air sorting tube part 11 to be 1500 cm^2 , and the axial length of the second air sorting tube part 11 was 100 cm.

The diameter of the tubular body of the second air sorting tube part 11 was large enough to allow the opening area of the tubular body of the second air sorting tube part 11 to be 3.3 times larger than the opening area of the tubular body of the first air sorting tube part 2.

[0045]

The polycrystalline silicon rod obtained by the Siemens process was crushed with use of a raw material polycrystalline silicon block crushing device and a hammer made of tungsten carbide, so as to obtain a crushed polycrystalline silicon mixture 4, which was in turn conveyed by the feeder 5 to the inlet 3 for the crushed polycrystalline silicon mixture that was provided in an intermediate height position of the first air sorting tube part 2 and was then introduced therinto. The crushed polycrystalline silicon mixture was conveyed to the inlet 3 at a speed of 10 m/min.

[0046]

Here, the crushed polycrystalline silicon mixture 4 contained the chunk material in an amount of 92% by mass, the chip material in an amount of 3% by mass, the powder material in an amount of 5% by mass, and dust in a slight amount. The mixture of these crushed materials had a surface metal concentration of 4 ppbw.

[0047]

In the air sorting device 1, the operation of the dust collector 10 applies a suction force to the inside of the tubular body of the first air sorting tube part 2. The suction force was strong enough to achieve a wind power of 13 m/s in a range from 15 cm below a lower end of the opening of the inlet 3 for the crushed polycrystalline silicon mixture to 15 cm above an upper end of the opening of the inlet. Therefore, the crushed polycrystalline silicon mixture 4 that had been introduced into the first air sorting tube part 2 was sorted into a chunk material 6 and a small particle diameter material 7. The chunk material 6 fell downwards and was taken out of the vent 8 for the chunk material at a lower end of the first air sorting tube part 2. An amount in which it was taken out accounted for 98% by mass of the chunk material 6 contained in the crushed polycrystalline silicon mixture 4 subjected to the sorting. This was a good collecting result. Further, the surface metal concentration of the chunk material 6 taken out was measured. The result was 2.5 ppbw, which was clean.

20 [0048]

The small particle diameter material 7 contained in the crushed polycrystalline silicon mixture 4 was sucked upwards in the first air sorting tube part 2 and flowed in the flow tube path 9 for the small particle diameter material. The

small particle diameter material 7 then entered the second air sorting tube part 11 from the inflow port 12 for the small particle diameter material. The sucking by the dust collector 10 applied a load that achieves a wind power of 5 m/s to the inside of the tubular body of the second air sorting tube part 11 in a range from 15 cm below a lower end of the opening of the inflow port 12 for the small particle diameter material to 15 cm above an upper end of the opening of the inlet.

[0049]

10 Therefore, the crushed polycrystalline silicon mixture 4 that had been introduced into the second air sorting tube part 11 was sorted into the chip material 13 and the powder material 14. The chip material 13 fell downwards and was taken out of a vent 15 for the chip material at the lower end of the second air sorting tube part 11. An amount in which it was taken out accounted for 83% by mass of the chip material 13 contained in the crushed polycrystalline silicon mixture subjected to the sorting. This was a good collecting result.

20 [0050]

Note that in this second air sorting tube part 11, substantially all of the remaining chunk material 6 that had not been sufficiently sorted by the first air sorting tube part 2 and was left in the flow tube path 9 for the small particle

diameter material fell downwards and was taken out of the vent 15 for the chip material together with the chip material 13.

[0051]

5 The powder material 14 contained in the small particle diameter material 7 was sucked upwards in the second air sorting tube part 11 together with the dust and flowed in the flow tube path 9 for the small particle diameter material again, followed by being collected by the filter 19 of the dust
10 collector 10. The powder material (including the dust) collected by the filter 19 was removed from the filter 19 and was taken out of the powder material vent 17 of the dust collector 10, followed by being collected in a bucket provided below. The amount in which the powder material (including
15 dust) 14 was taken out accounted for 99% by mass of the powder material 14 contained in the crushed polycrystalline silicon mixture subjected to the sorting. This was a good collecting result.

[0052]

20 Comparative Example 1

The crushed polycrystalline silicon mixture 4 was sorted by particle size with use of a circular vibrating sieve device. The members of the above circular vibrating sieve device were made of stainless steel lined with resin. The

sieving was performed with use of each of sieves having sieve openings of 500 μm , 1,000 μm , and 1,500 μm , in decreasing order of the sieve openings of the sieves.

[0053]

5 After the sieving, a surface metal concentration of the chunk material obtained as the material remaining on the sieve having a sieve opening of 1,500 μm was measured. The result was 12.3 ppbw, which was greater than the result in Example 1 above.

10 [0054]

Comparative Example 2

With use of the same device as the polycrystalline silicon block crushing device used in Example 1 of Patent Literature 2, from its top surface opening into its falling
15 moving part, the same crushed polycrystalline silicon mixture as the one that was introduced into the inlet 3 for the crushed polycrystalline silicon mixture of the first air sorting tube part 2 in Example 1 above was caused to fall. At that time, in the falling moving part, air is horizontally
20 sucked from the sucking removal part 40 by the same sucking amount of 10 m/s as the upward suction force in the vicinity of the height in which the inlet 3 for the crushed polycrystalline silicon mixture is formed, in the first air sorting tube part 2 in Example 1 above.

[0055]

However, as a result, most of what was sucked by the sucking removal part was the powder material, whereas the chip material was sorted in only an amount corresponding to 15% by mass of the chip material 13 contained in the crushed polycrystalline silicon mixture, and most of the chip material had fallen onto the lower receiver together with the chunk material.

[0056]

10 The present invention is not limited to the description of the embodiments above, but may be altered in various ways by a skilled person within the scope of the claims. The present invention encompasses, in its technical scope, any configuration based on an appropriate combination of
15 technical means disclosed in different embodiments.

Reference Signs List

[0057]

- 1 Air sorting device
- 20 2 First air sorting tube part
- 3 Inlet for crushed polycrystalline silicon mixture
- 4 Crushed polycrystalline silicon mixture
- 5 Feeder
- 6 Chunk material

- 7 Small particle diameter material
- 8 Vent for chunk material
- 9 Flow tube path for small particle diameter material
- 10 Dust collector
- 5 11 Second air sorting tube part
- 12 Inflow port for small particle diameter material
- 13 Chip material
- 14 Powder material
- 15 Vent for chip material
- 10 17 Powder material vent
- 18 Exhaust port
- 19 Filter

Claims

[Claim 1]

A crushed polycrystalline silicon mixture air sorting device comprising:

5 a first air sorting tube part that includes a tubular body provided so as to stand vertically and that is provided with an inlet for a crushed polycrystalline silicon mixture containing chunk material and small particle diameter material having a smaller particle diameter than that of the
10 chunk material, the inlet being formed in an axially middle area of the tubular body, the first air sorting tube part being configured to perform air sorting between the chunk material and the small particle diameter material, wherein

 the chunk material in the crushed polycrystalline
15 silicon mixture that has been introduced into the tubular body falls downwards, and

 a flow tube path for the small particle diameter material is connected to an upper end of the first air sorting tube part and is provided with a sucking section located
20 upstream of the flow tube path, the sucking section being configured to apply a suction force for sucking the small particle diameter material upwards.

[Claim 2]

The crushed polycrystalline silicon mixture air sorting device according to claim 1, in which a second air sorting tube part that includes a tubular body provided so as to stand vertically midway in the flow tube path and that is
5 provided with an inflow port for the small particle diameter material from the flow tube path is interposed, the inflow port being formed in an axially middle area of the tubular body,

the tubular body having a diameter adjusted so as to
10 apply a suction force that allows chip material in the small particle diameter material that has entered the tubular body to fall downwards and that sucks powder material having a smaller particle diameter than that of the chip material upwards, the second air sorting tube part being configured
15 to perform air sorting between the chip material and the powder material.

[Claim 3]

A method for manufacturing chunk material from a
20 crushed polycrystalline silicon mixture, the method comprising:

introducing, with use of the crushed polycrystalline silicon mixture air sorting device according to claim 1, a

crushed polycrystalline silicon mixture into the tubular body of the first air sorting tube part;

sorting the crushed polycrystalline silicon mixture by air; and

5 collecting the chunk material from a lower end of the tubular body.

[Claim 4]

A method for manufacturing chip material from a
10 crushed polycrystalline silicon mixture, the method comprising:

using the crushed polycrystalline silicon mixture air sorting device according to claim 2 to sort, by air, the small particle diameter material that has entered the tubular body
15 of the second air sorting tube part; and

collecting the chip material from a lower end of the tubular body.

[Claim 5]

20 A method for manufacturing powder material from a crushed polycrystalline silicon mixture, the method comprising:

using the crushed polycrystalline silicon mixture air sorting device according to claim 2 to sort, by air, the small

particle diameter material that has entered the tubular body of the second air sorting tube part; and

collecting the powder material that has exited from an upper end of the tubular body to the flow tube path.