

SPECIFICATIONTITLE OF THE INVENTION

SILICA GLASS CRUCIBLE

5 AND METHOD FOR MANUFACTURING THE SAME

TECHNICAL FIELD

[0001]

The present invention is related to a silica glass
10 crucible used for pulling up a silicon crystal and a
method for manufacturing the silica glass crucible. In
particular, the present invention is related to a silica
glass crucible which has a high degree of roundness and
which is used for pulling up a silicon single crystal
15 used as a semiconductor material or a polycrystalline
silicon used as a solar cell material.

BACKGROUND OF THE INVENTION

[0002]

20 A silica glass crucible is used for pulling up a
silicon single crystal such as semiconductor material
or a polycrystalline silicon such as a solar cell. For
example, single crystal silicon is mainly manufactured
by a method in which a silicon melt is produced by melting
25 a polycrystalline silicon nugget which has been charged

in a silica glass crucible, dipping a seed crystal in the silicon melt and pulling up the seed crystal. The silicon crystal used for a solar cell material is manufactured by the same pulling up method but has a lower yield of single crystallization than the silicon single crystal used for a semiconductor material.

[0003]

The silicon crystal is pulled up while applying a heat uniformly to the silicon melt by rotating the silica glass crucible. In this case, if the roundness (level of roundness compared to a perfect circle) of the interior surface and exterior surface of the crucible is low, because the level of vibration of the silicon melt becomes large due to horizontal swaying when rotating the crucible, the oxygen in-plane distribution becomes non-uniform and the yield of crystallization decreases. Therefore, it is preferable that the silica glass crucible used for pulling up silicon crystal be as close to a perfect circle as possible.

[0004]

A rotation mold method is known as one manufacturing method of a silica glass crucible. In this method, the crucible is manufactured by depositing silica powder to a predetermined thickness on inner surface of a crucible shaped rotation mold and this layer

of silica powder is heated and melted while rotating the mold to be vitrified (Japanese Patent Applications Laid-open Nos. S56-17996 and S56-149333). In addition, a manufacturing method of a silica glass crucible is also known in which silica powder which is partially melted is attached to the interior surface of a rotation mold (Japanese Patent Application Laid-open No. H01-148718).

[0005]

10 In the method for manufacturing a silica glass crucible stated above, when the thickness of a silica powder layer or silica glass layer which is attached to the interior surface of a mold is uneven, the roundness of the interior surface of the glass crucible is reduced and the yield of crystallization can not be increased 15 when pulling up the silicon crystal. In addition, because the horizontal swaying becomes larger when rotating the crucible, the yield of crystallization is reduced even when the roundness of the exterior surface 20 of the crucible is low.

SUMMARY OF THE INVENTION

[0006]

The present invention solves the conventional 25 problems described above with regard to a silica glass

crucible used for pulling up silicon crystal, and shows the standard of roundness of a silica glass crucible for achieving a fixed yield or above of crystallization of a silicon crystal. More specifically, the present
5 invention shows a permitted range of roundness of the silica glass crucible for obtaining a crystallization yield of 80% or more.

[0007]

The present invention has been achieved to solve
10 the conventional problem, a silica glass crucible for pulling up a silicon crystal according to the present invention has a roundness S_x of an interior surface of the silica glass crucible and a roundness S_y of an exterior surface of the silica glass crucible in at least
15 a wall part of the silica glass crucible both being 0.4 or less ($S_x / M \leq 0.4$, $S_y / M \leq 0.4$) to a maximum thickness M in the same measurement height as the roundness.

[0008]

In at least a wall part of the silica glass crucible,
20 because the roundness S_x of the interior surface of the crucible and the roundness S_y of the exterior surface of the crucible is 0.4 or less ($S_x / M \leq 0.4$, $S_y / M \leq 0.4$) to the maximum thickness M in the same measurement height as the roundness, when the crucible is used to
25 pull up the silicon crystal, the oxygen in-plane

distribution of the silicon crystal is uniform and a crystallization of more than 80% can be obtained.

[0009]

Furthermore, the silica glass crucible for pulling
5 up a silicon crystal according to the present invention
has a distance L between the center of an interior
surface of the silica glass crucible and the center of
an exterior surface of the silica glass crucible in at
least a wall part being 0.01 or less ($L \leq 0.01D$) of the
10 longest exterior diameter D of the silica glass crucible.
Because the distance L between the center of the interior
surface and the center of the exterior surface of the
crucible is 0.01 or less ($L \leq 0.001 D$) of the longest
diameter D of the exterior surface of the crucible, the
15 oxygen in-plane distribution of the crystal is uniform
and a crystallization of more than 80% can be obtained.

[0010]

In addition, a manufacturing method of the silica
glass crucible according to the present invention
20 includes depositing silica powder on an interior surface
of a rotating crucible shaped mold, vitrifying by
heating at a high temperature a layer of the silica
powder while the mold rotates, and controlling an amount
of horizontal sway of the interior surface of the mold
25 to 0.1% or less of an interior diameter of the mold.

According to the manufacturing method of the present invention, when manufacturing a silica glass crucible by a rotation mold method, by placing a dial gage on the exterior surface of the mold and controlling the amount of horizontal sway of the interior surface to 0.1% or less of the internal diameter of the mold, it is possible to maintain the roundness (S_x ratio and S_y ratio to M , L ratio to D) of the silica glass crucible within the range stated above.

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BRIEF DESCRIPTION OF THE DRAWINGS

[0011]

The above and other objects, features and advantages of this invention will become more apparent by reference to the following detailed description of the invention taken in conjunction with the accompanying drawings, wherein:

FIG. 1 is longitudinal cross sectional view of a silica glass crucible;

FIG. 2 is a horizontal cross sectional view of a silica glass crucible;

FIG. 3 is a diagram which explains the roundness of a crucible interior surface;

FIG. 4 is a diagram which explains an axis misalignment between the center of an interior surface

and the center of an exterior surface; and

FIG. 5 is a flowchart which shows the manufacturing method of a silica glass crucible of the present invention.

5

DETAILED DESCRIPTION OF THE EMBODIMENTS

[0012]

The present invention will be explained in detail below based on the embodiments. FIG. 1 is a longitudinal cross sectional view of a silica glass crucible. As shown in FIG. 1, the silica glass crucible 10 of the present invention is used for pulling up a silicon crystal and includes a cylindrical wall part 10A, a bottom part 10B arranged below the wall part 10A, and a corner part 10C positioned between the wall part 10A and the bottom part 10B. In at least the wall part 10A of the silica glass crucible 10 of the present invention, the roundness S_x of the interior surface of the crucible and the roundness S_y of the exterior surface of the crucible are 0.4 or less ($S_x / M \leq 0.4$, $S_y / M \leq 0.4$) to the maximum thickness M in the same height as the height measured for the roundness S_x and S_y .

[0013]

FIG. 2 is a horizontal cross sectional view of a crucible wall part (side wall) when the silica glass

crucible shown in FIG. 1 is cut at an arbitrary position shown by the dashed line. Furthermore, the roundness of the interior surface 11 of the crucible in FIG. 2 is shown in FIG. 3. When the interior surface 11 is sandwiched by inner and outer concentric circles (perfect circles) A, B as is shown in FIG. 3, the roundness S_x of the interior surface 11 is expressed by a semi-diameter correction between the circle A and circle B when the interval between these concentric circles A, B is at its smallest. Circle A contacts with a part of the crucible interior surface 11 which projects the furthest outward, and circle B contacts with a part of the crucible interior surface 11 which projects the furthest inward. Although not shown in the diagram, the same as the crucible interior surface 11, when the exterior surface 12 is sandwiched by inner and outer concentric circles, the roundness S_y of the crucible exterior surface 12 is expressed by a semi-diameter correction when the interval between these concentric circles is at its smallest.

[0014]

The roundness S_x of the crucible interior surface 11 and the roundness S_y of the crucible exterior surface 12 of the silica glass crucible 10 of the present invention are 0.4 or less ($S_x / M \leq 0.4$, $S_y / M \leq 0.4$)

to (refer to FIG. 2) the maximum thickness M of the crucible wall part which is measured by the same height as the roundness.

[0015]

5 If the roundness S_x of the crucible interior surface 11 or the roundness S_y of the crucible exterior surface 12 is large, the thickness difference of the crucible wall part becomes larger and when the crucible is rotated because the horizontal sway of the silicon
10 melt which is charged in the crucible becomes larger when the crucible is rotated. It is therefore preferable that the roundness S_x and S_y are as small as possible. The interval between the crucible interior surface 11 and the crucible exterior surface 12 is the thickness
15 of the crucible and the roundness S_x of the interior surface and the roundness S_y of the exterior surface are related to the thickness of the crucible.

[0016]

 According to the present invention, if the
20 roundness S_x of the interior surface and the roundness S_y of the exterior surface are controlled to less than 0.4 to the maximum thickness M of the crucible wall part 10A in the same measurement height as the roundness, it is possible to make the oxygen in-plane distribution
25 within the crystal uniform and also achieve a high yield

of crystallization.

[0017]

In manufacturing the silica glass crucible, although the roundness S_x of the crucible interior surface and the roundness S_y of the crucible exterior surface are made as small as possible to approach a perfect circle, in the actual manufacturing process it is difficult to produce a perfect circle. Therefore, it is a great advantage and discovery that it is sufficient to control the roundness S_x of the crucible interior surface and the roundness S_y of the crucible exterior surface to within the above stated conditional range by the present invention.

[0018]

Because the fluid level of the silicon melt which is charged in the silica glass crucible gradually decreases from the side wall part (wall part) to the corner part by pulling up the silicon crystal, the crucible interior surface roundness S_x and the crucible exterior surface roundness S_y are expected to meet the above stated conditions ($S_x / M \leq 0.4$, $S_y / M \leq 0.4$) in at least the wall part of the crucible.

[0019]

In addition, as shown in FIG. 4, in the silica glass crucible used for pulling up a silicon crystal, when the

distance (misaligned axis) L , from the center of the interior surface of the crucible to the center of the exterior surface of the crucible is large, the horizontal sway of the crucible when it is rotated becomes large. It is therefore preferred that this misaligned axis is made as small as possible. However, it is difficult to completely eliminate the misaligned axis in the actual manufacturing process.

[0020]

Here, according to the present invention, if the misaligned axis L stated above is 0.01 or less ($L \leq 0.01 D$), or more preferably 0.005 or less ($L \leq 0.005 D$) of the longest diameter D which passes through the center of the crucible exterior surface, it is possible to make the oxygen in-plane distribution within the crystal uniform and also achieve a high yield of crystallization when pulling up the silicon crystal. Because it is difficult to completely eliminate the misaligned axis in the actual manufacturing process, it is a great advantage and discovery that it is sufficient to control the misaligned axis to within the above stated conditional range.

[0021]

In manufacturing the silica glass crucible by depositing silica powder on the interior surface of a

rotating crucible shaped mold and vitrifying by heating this silica powder layer under a high temperature while the mold is rotating, the silica glass crucible of the present invention can be manufactured
5 by placing a dial gage on the interior surface of the mold and controlling the amount of horizontal sway of this interior surface to less than 0.1% of the mold's interior diameter.

[0022]

10 FIG. 5 is a flowchart which shows a manufacturing method of the silica glass crucible of the present invention. In the manufacture of the silica glass crucible, first the amount of horizontal sway of the interior surface of the mold is adjusted while rotating
15 the mold (step S11). At this time, the amount of horizontal sway is adjusted to less than 0.1% of the interior diameter of the mold by using a dial gage. Next, silica powder is deposited on the interior surface of the rotating mold and a silica powder layer is formed
20 (step S12). Following this, vitrification is performed by heating the silica powder layer within the rotating mold at a high temperature and a silica glass crucible which has the basic shape shown in FIG. 1 is formed (step S13). In a silica glass crucible manufactured in this
25 way, because the crucible interior surface roundness S_x

and the crucible exterior surface roundness S_y meet the above stated conditions ($S_x / M \leq 0.4$, $S_y / M \leq 0.4$) in a least the wall part of the crucible, it is possible to make the oxygen in-plane distribution within the crystal uniform and also achieve a high yield of crystallization when pulling up the silicon crystal.

[0023]

The present invention is in no way limited to the aforementioned embodiments, but rather various modifications are possible within the scope of the invention as recited in the claims, and naturally these modifications are included within the scope of the invention.

[0024]

For example, the silica glass crucible of the present invention is effective for pulling up a silicon single crystal used as because it is possible to achieve a high yield of crystallization. However, the silica glass crucible of the present invention is also effective for pulling up a polycrystalline silicon used as a solar cell material because it is possible to make the oxygen in-plane distribution within the crystal uniform.

[0025]

(Examples)

The present invention is shown in detail using examples and comparative examples below.

[0026]

(Examples 1 - 4)

5 A silica glass crucible (32 inch interior diameter) of the present invention was manufactured and a silicon single crystal was pulled up under the conditions shown in Table 1. At this time, the silicon single crystal was pulled up while rotating the silica glass crucible
10 so that the silicon melt was uniformly heated and the oxygen in-plane distribution within the crystal became as uniform as possible. The rotation speed of the silica glass crucible was 5 rpm. Next, the crystallization yield and oxygen in-plane distribution within silicon
15 single crystal which was pulled up was measured.

[0027]

The oxygen in-plane distribution within the crystal was measured from four points of oxygen density in a radial direction which includes the center of a
20 wafer cut from a silicon ingot. Usually, the oxygen density decreases gradually in a radial direction from the center of the wafer. However, this reduced amount is particularly noticeable near the periphery of the wafer. Therefore, the points to be measured are usually
25 located at (1) center of the wafer, (2) a position $r/2$

(r is a radius) from the center of the wafer, (3) a position 10 mm from the periphery of the wafer, and (4) a position 5 mm from the periphery of the wafer. The oxygen in-plane distribution of a wafer was evaluated in the following way. In the case where all the measured values of these four points are within a predetermined range, more specifically, in the case where the all the measured values are within $10\% \pm$ of the oxygen density at the center of the wafer, the oxygen in-plane distribution within the crystal is considered [uniform], and in the case where these values are not within this range, the oxygen in-plane distribution within the crystal is considered [non-uniform]. For example, when the oxygen density at the center of the wafer is 12.55×10^{17} atms / cm^3 , if another measured value is within the range $11.30 - 13.80 \times 10^{17}$ atms / cm^3 , then the oxygen density is uniform. Furthermore, the oxygen density within the crystal was measured by a Fourier transform infra-red spectrophotometer (commonly called FTIR).

[0028]

The yield of crystallization was measured by the weight of the straight body part of the silicon single crystal which was pulled up divided by the weight of silicon material put into the crucible. That is,

because not all of the silicon melt within the crucible is consumed and only the straight body part excluding the top part and tail part of a silicon ingot is used in the calculation of the yield of crystallization. Even
5 if a sufficient silicon single crystal is pulled up, the crystallization yield is below 100% and favorable if more than 80%. The results of the yield of crystallization of the silicon crystal and oxygen in-plane distribution within the crystal are shown in
10 Table 1.

[0029]

(Comparative Examples 1 - 3)

A silica glass crucible with a 32 inch interior diameter was manufactured and a silicon single crystal
15 was pulled up under the conditions shown in Table 1. Next, the same as in the example stated above, the crystallization yield and oxygen in-plane distribution within silicon single crystal which was pulled up was measured. The results are shown in Table 1.

20 [0030]

(Table 1)

	Interior surface roundness S_x	Exterior surface roundness S_y	Maximum thickness M	Roundness ratio		Oxygen in-plane Distribution	Crystallization yield (%)
				Interior Surface	Exterior Surface		
Example 1	1.5mm	1.7mm	15mm	0.10	0.11	O	88
Example 2	5.3mm	5.7mm	15mm	0.35	0.38	O	83
Example 3	6.0mm	3.0mm	15mm	0.40	0.20	O	80
Example 4	3.0mm	6.0mm	15mm	0.20	0.40	O	81
Comparative Example 1	6.8mm	2.1mm	15mm	0.45	0.14	X	52
Comparative Example 2	1.8mm	7.2mm	15mm	0.12	0.48	X	47
Comparative Example 3	7.1mm	7.7mm	15mm	0.47	0.51	X	51

(note) roundness ratio is a ratio of the roundness to the maximum thickness M in the same measurement height as the roundness.

"O" indicates that the oxygen in-plane distribution is uniform and "X" indicates non-uniform.

[0031]

(Examples 11 - 13)

A silica glass crucible (32 inch interior diameter) of the present invention was manufactured and a silicon single crystal was pulled up under the conditions shown in Table 2. Next, the same as in the example and comparative example stated above, the crystallization yield and oxygen in-plane distribution within the silicon single crystal was measured. The results are shown in Table 2.

[0032]

(Comparative Examples 11 - 12)

A silica glass crucible with a 32 inch interior diameter was manufactured and a silicon single crystal was pulled up under the conditions shown in Table 2. Next, the same as in the example and comparative example

stated above, the crystallization yield and oxygen in-plane distribution within the silicon single crystal was measured. The results are shown in Table 2.

[0033]

5 (Table 2)

	Axis misalignment L between interior surface and exterior surface	Longest diameter D of exterior surface	Axis misalignment ratio	Oxygen in-plane Distribution	Crystallization yield (%)
Example 11	4.1mm	825mm	0.005	O	88
Example 12	5.6mm	825mm	0.007	O	83
Example 13	8.3mm	825mm	0.010	O	80
Comparative Example 11	9.9mm	825mm	0.012	X	65
Comparative Example 12	12.4mm	825mm	0.015	X	43

(note) Axis misalignment ratio is a ratio of the axis misalignment L to the longest diameter D of the exterior surface.

[0034]

As is shown in Tables 1 and 2, in the silica glass crucible of the present invention the oxygen in-plane distribution within the silicon single crystal was uniform and the yield of crystallization was high. On the other hand, in the silica glass crucible in the comparative examples the oxygen in-plane distribution within the silicon single crystal was non-uniform and the yield of crystallization was low. In addition, as is shown in examples 11 to 13, the yield of crystallization was at its highest when the axis misalignment ratio was 0.01 or less, and the larger the

axis misalignment the lower the yield of crystallization became. Particularly, as is shown in example 11, the yield of crystallization was 88% when the axis misalignment ratio was 0.005 and the lower the axis
5 misalignment the higher the yield of crystallization became.

What is claimed is:

1. A silica glass crucible for pulling up a silicon crystal having a roundness S_x of an interior surface of the silica glass crucible and a roundness S_y of an exterior surface of the silica glass crucible in at least a wall part of the silica glass crucible both being 0.4 or less ($S_x / M \leq 0.4$, $S_y / M \leq 0.4$) to a maximum thickness M in the same measurement height as the roundness.

2. A silica glass crucible for pulling up a silicon crystal comprising:

a cylindrical wall part;
a bottom part arranged below the wall part; and
a corner part arranged between the wall part and the bottom part; and

wherein

at a certain height of at least the wall part, when a roundness of an interior diameter of the silica glass crucible is S_x , a roundness of an exterior diameter of the silica glass crucible is S_y , and a maximum thickness of the silica glass crucible is M , S_x and S_y are both 0.4M or less ($S_x \leq 0.4M$, $S_y \leq 0.4M$).

3. The silica glass crucible according to claim 2, wherein at the certain height when L is a distance from the center of the interior diameter of the silica glass crucible to the center of the exterior diameter of the silica glass crucible, and D is the largest exterior diameter of the silica glass crucible, L is 0.01 or less ($L \leq 0.01D$).

4. A silica glass crucible for pulling up a silicon crystal having a distance L between the center of an interior surface of the silica glass crucible and the center of an exterior surface of the silica glass crucible in at least a wall part being 0.01 or less ($L \leq 0.01D$) of the longest exterior diameter D of the silica glass crucible.

5. A method for manufacturing a silica glass crucible comprising:

depositing silica powder on an interior surface of a rotating crucible shaped mold;

vittrificating by heating at a high temperature a layer of the silica powder while the mold rotates; and

controlling an amount of horizontal sway of the interior surface of the mold to 0.1% or less of an interior diameter of the mold.

6. A method for manufacturing a silica glass crucible comprising:

adjusting an amount of horizontal sway of an
5 interior surface of a mold to 0.1% or less of an interior diameter of the mold, the horizontal sway being generated when the mold having a crucible shape is rotated at a predetermined speed;

depositing silica powder on the interior surface
10 of the rotating mold in which the horizontal sway is adjusted; and

vittrificating a layer of the silica powder by heating at a high temperature while the mold rotates.