

SILICON SINGLE CRYSTAL PRODUCTION METHOD

TECHNICAL FIELD

5 The present invention relates to a production method of a silicon single crystal. More specifically, the present invention relates to a production method of a silicon single crystal based on the Czochralski method (hereinafter referred to as "CZ method") which can prevent occurrence of the dislocations during the separation
10 process from the silicon melt, thereby improving the shape of the separation surface of the produced silicon single crystal.

BACKGROUND ART

The CZ method includes steps of heating raw material polycrystalline silicon in a crucible to produce a raw material melt,
15 immersing a seed crystal in the raw material melt, and finally lifting up the produced silicon single crystal by gradually rewinding a wire attached to the seed crystal.

The method further includes growing a cone section with a gradually-extending cone diameter, i.e. an extending-diameter
20 section, which stretches from the contact surface with the seed crystal, and then growing a straight-body section, i.e. a constant-diameter section, when the crystal diameter has reached a target diameter. The silicon single crystal is finally separated
25 from the raw material melt when the length of the straight-body section reaches a predetermined length.

In a conventional production method, the step of separating the silicon single crystal from the raw material melt tends to cause a sudden temperature drop at the lower end of the silicon single
30 crystal, which is the separation surface from the silicon melt. This may result in a considerably-reduced crystallization ratio due to the occurrence of the slip dislocations within the silicon single crystal after being lifted up.

As a countermeasure to this, an conventional production method
35 further includes a step of gradually narrowing the diameter of the silicon single crystal after the growth of the straight-body section so that the contact surface between the silicon single crystal and the raw material melt will be small enough to avoid the occurrence

of the dislocations, before separating the silicon single crystal from the raw material melt. This narrowed section is generally called a tail section.

5 However, the tail section is known to cause a decreased yield ratio since its crystal diameter is smaller than a desired value, and therefore this section is not considered to be a product.

Accordingly, the step of producing the tail section should preferably be reduced or even omitted in order to increase productivity of the silicon single crystal.

10 There already exist a number of techniques for reducing or omitting the generation step of the tail section, and one of them includes steps of lifting up a crucible containing the raw material melt at the same lifting speed as that of the silicon single crystal after the growth of the straight-body section, and rendering the bottom
15 surface of the silicon single crystal protruding downward, and suppresses the occurrence of the dislocations during the separation of the silicon single crystal from the raw material melt, accordingly (See the Japanese Patent Application Publication No. 2007-176761).

In recent years, the silicon wafer diameter is becoming larger
20 and larger, and 300 mm wafer now is the main product. The production of a silicon single crystal with a large-diameter generally involves a strengthened natural convection current within the crucible due to the increased weight of the silicon melt, which tends to cause dislocations and a deformation of the silicon single crystal.

25 Therefore, various approaches have been made to control the natural convection current by means of applying a horizontal magnetic field to the silicon melt, for the purpose of suppressing the dislocations and the deformation.

However, application of the horizontal magnetic field adversely
30 causes a decreased temperature difference between the center and the periphery of the phase boundary of the silicon single crystal, compared to its absence, which can prevent the silicon single crystal from forming a downward convex shape on the phase boundary, and therefore disrupts the omission of the tail section intended to
35 suppress the occurrence of the dislocations during the separation of the silicon single crystal from the melt, while achieving the suppression of the dislocations and the deformation in the straight-body section.

In order to solve the above-mentioned problems, the inventors of the present invention performed intensive studies, and then worked out a production method allowing for the transformation of the whole growth front of the silicon single crystal faced with the melt, into a convex shape protruding in the opposite direction to the lifting direction of the silicon single crystal, even under the influence of the horizontal magnetic field.

The inventors finally achieved the present invention when they learned that the separation of the silicon single crystal from the melt taking place after the transformation into the convex shape will allow for the suppression of the dislocations, and hence the successful omission of the tail section.

SUMMARY

The inventors performed intensive studies especially in pursuit of a crystal growth method for achieving the separation of the silicon single crystal from the melt at a high success rate as well as the suppression and the prevention of the dislocations.

As a result, they finally achieved the suppression of the dislocations and the successful omission of the tail section by growing the straight-body section of the silicon single crystal under the influence of the horizontal magnetic field with a magnetic flux density at its magnetic center being equal to or more than 1000 Gauss, and equal to or less than 2000 Gauss; reducing the relative lifting speed of the silicon single crystal to the surface of the melt to 0 mm/minute maintaining the static state until there is a decrease in the apparent weight of the silicon single crystal; further maintaining the static state so that the whole growth front of the silicon single crystal faced with the melt will form a convex shape protruding in the direction opposite to the lifting direction of the silicon single crystal; and finally separating the silicon single crystal from the melt.

According to an aspect of the present invention, there is provided a production method of a silicon single crystal based on the Czochralski method, comprising steps of:

5 (1) growing a straight body section of said silicon single crystal by lifting up said silicon single crystal;

 (2) reducing the relative lifting speed of said silicon single crystal to the surface of said melt to 0 mm/minute, after
10 said step (1);

 (3) measuring the apparent weight of the silicon single crystal and maintaining the static state where the relative lifting speed of said silicon single crystal is kept at 0
15 mm/minute until there is a decrease in the apparent weight of said silicon single crystal, after said step (2);

 (4) further maintaining said static state so that the whole growth front of said silicon single crystal faced with
20 said melt will form a convex shape protruding in the opposite direction to the lifting direction of said silicon single crystal, after said step (3), wherein a horizontal magnetic field is applied to a raw material melt contained in a crucible at least during steps (1) to (4), the horizontal magnetic field
25 having a magnetic flux density at its magnetic center which is equal to or more than 1000 Gauss, and equal to or less than 2000 Gauss; and

 (5) separating said silicon single crystal from said
30 melt, after said step (4).

EFFECT OF INVENTION

The present invention allows for an appropriate control of the natural convection current in the melt and consequently
35 the suppression of the deformation and the dislocations in the straight-body section, by lifting up the produced silicon single

crystal under the influence of the horizontal magnetic field with a magnetic flux density at its magnetic center being equal to or more than 1000 Gauss, and equal to or less than 2000 Gauss.

5 The present invention also allows for the suppression of the dislocations and consequently the successful omission of the tail section, by suspending the relative movement of the silicon single crystal to the surface of the silicon melt until there is a decrease in its apparent weight, and maintaining the static state so that the while growth face of the silicon single crystal will form a convex
10 shape protruding in the opposite direction to the lifting direction of the silicon single crystal.

BRIEF DESCRIPTION OF THE DRAWINGS

Fig.1 is a schematic view of a silicon single crystal production
15 equipment according to an embodiment of the present invention.

Fig. 2 is a flowchart showing a production method of a silicon single crystal according to the embodiment.

Fig. 3 is a partially-enlarged schematic view showing shape changes of the growth front of the silicon single crystal.

20 Fig. 4 is a picture showing an example of the growth front of the silicon single crystal whose tail-section has failed to be omitted.

Fig. 5 is a picture showing an example of the growth front of the silicon single crystal whose tail-section has been successfully omitted.

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DETAILED DESCRIPTION

An embodiment of the present invention will be described below with reference to the attached drawings. Referring to Fig. 1, the silicon single crystal production equipment 100 according to the
30 present embodiment produces a silicon single crystal 2 based on the CZ method. The silicon single crystal production equipment 100 includes a crucible 30 for containing melt 32, a driving unit 10 for not only lifting up/down and rotating the silicon single crystal 2 but also rotating the crucible 30, and a magnetic field generating
35 unit 50 for generating the magnetic field applicable to the melt 32.

The silicon single crystal production equipment 100 also includes a controller 1 for controlling not only the lifting movement and

the rotation of the silicon single crystal 2 and the rotation of the crucible 30 both driven by the driving unit 10, but also the horizontal magnetic field generated by the magnetic field generating unit 50.

5 The silicon single crystal production equipment 100 also includes a chamber 20 for containing the crucible 30 and the silicon single crystal 2, heaters 42 placed along the side wall of the crucible 30, heaters 44 placed under the crucible 30, and a heat insulator 26 attached to the side wall and the inner bottom of the chamber
10 20.

 The silicon single crystal production equipment 100 includes a wire 3 hung from the top of the chamber 20, a weight measuring unit 5 for measuring the apparent weight of the silicon single crystal 2, and a chuck 6 attached to the tip of the wire 3 for holding seed
15 crystal 8.

 The weight measuring unit 5 is typically a load cell. The apparent weight is calculated by subtracting the buoyancy of the melt 32 from the gross weight of the silicon single crystal 2.

 The silicon single crystal production equipment 100 also includes
20 a gas inlet 22 of the chamber 20 for taking in inert gas, a gas outlet 24 for discharging the inert gas from the chamber 20, and a rectifying cylinder 60 for rectifying the inert gas flow. The inert gas is typically Ar gas.

 The melt 32 is a melt of raw material polycrystalline silicon.
25 The crucible 30 is typically made of synthetic quartz glass. The base of the crucible 30 is connected with an axis 34 for supporting the crucible 30. The rectifying cylinder 60 is located above the crucible 30 and substantially forms a circular truncated cone.

 A driving unit 10 includes a winder 12 for winding up/down the
30 wire 3 and a crucible driver 14 for rotating the crucible 30.

 The winder 12 is capable of rewinding and rotating axially the wire 3 at the same time. The axis 34 is connected to the crucible driver 14, and the crucible 30 can be rotated by the crucible driver 14 via the axis 34.

35 The magnetic field generating unit 50 includes a pair of coils 52. The pair of coils 52 is placed outside the chamber 20 to sandwich the chamber 20. The magnetic center L2 of the horizontal magnetic field generated by the coils 52 is substantially perpendicular to

the central axis L1 of the silicon single crystal 2.

The magnetic field generating unit 50 is capable of moving up and down along the central axis L1 of the silicon single crystal 2 so that the position of the magnetic center L2 will be shifted along
5 the central axis L1 of the silicon single crystal 2.

The controller 1 is electronically connected to the winder 12, the heaters 42, 44, the crucible driver 14, and the magnetic field generating unit 50.

The controller 1 is capable of controlling the rewinding speed
10 of the wire 3, the rotation direction of the wire 3, and the rotation speed of the wire 3, i.e. the lifting speed of the silicon single crystal 2, the rotation direction of the silicon single crystal 2, and the rotation speed of the silicon single crystal 2 - by means of the winder 12.

15 The controller 1 is also capable of controlling the rotation direction and the rotation speed of the crucible 30 by means of the crucible driver 14.

The controller 1 is also capable of controlling the position of the coils 52 along the central axis L1 of the silicon single crystal
20 2, i.e. the position of the magnetic center L2 of the horizontal magnetic field.

The controller 1 is also capable of controlling the magnetic flux density at the magnetic center L2 of the horizontal magnetic field so that it will be equal to or more than 1000 Gauss, and equal to
25 or less than 2000 Gauss.

The controller 1 is also capable of controlling the output power of the heaters 42, 44, and controlling the temperature of the melt 32 by adjusting the output power of the heaters 42 with the output power of the heaters 44 kept constant.

30 The controller 1 is electronically connected to the weight measuring unit 5, and is capable of monitoring the apparent weight of the silicon single crystal 2.

The controller 1 mainly comprises a CPU and a memory device, and is capable of storing the system operations of the whole silicon
35 single crystal production equipment 100 into the memory device.

The controller 1 is typically a PC or an EWS (Engineering Work Station). The silicon single crystal production method described below can be implemented by the controller 1 for controlling the

operation of each unit.

The following is a production method of a silicon single crystal according to the present embodiment.

Referring to Fig. 2, the production method according to the present
 5 embodiment is to produce a silicon single crystal based on the CZ method, and includes an inert gas filling step S1 of creating an inert gas atmosphere inside the chamber 20; a melt producing step S2 of generating the melt 32; and a straight-body section growing
 10 step S3 (or step (1)) of growing the straight-body section 4 of the silicon single crystal 2. The production method also includes a suspension step S4 (or step (2)); a first static-state maintaining step S5 (or step (3)); a second static-state maintaining step S6
 15 (or step (4)); and a separating step S7 (or step (5)) of separating the silicon single crystal 2 from the melt 32, all of which are intended to omit the tail-section of the silicon single crystal.

The inert gas filling step S1 includes steps of sealing the chamber 20, and taking in the inert gas through the gas inlet 22 in parallel with evacuating the chamber 20 through the gas outlet 24 using a vacuum pump (not shown) in order to create the inert gas atmosphere
 20 inside the chamber 20.

The inert gas atmosphere inside the chamber 20 is maintained by an inert gas flow into the chamber 20 in the steps afterwards.

The melt producing step S2 which follows the inert gas filling step S1 includes a step of generating the melt 32 by melting the
 25 raw material injected into the crucible 30, using the heaters 42, 44.

The straight-body section growing step S3 which follows the melt producing step S2 includes steps of growing the straight-body section 4 of the silicon single crystal 2 by lifting down the seed
 30 crystal 8 to immerse it under the surface of the melt, using the winder 12, and lifting up the silicon single crystal 2 from the melt 32 using the winder 12 under the influence of the horizontal magnetic field applied to the melt 32 which has a magnetic flux density at its magnetic center L2 being equal to or more than 1000 Gauss, and
 35 equal to or less than 2000 Gauss.

The silicon single crystal 2 and the crucible 30 rotate axially in the opposite directions to each other.

The straight-body section 4 of the silicon single crystal 2 is

a cylindrical portion with a nearly constant diameter in the direction parallel to the central axis L1 of the silicon single crystal 2. The straight-body section growing step S3 includes a step of growing the straight-body section 4 when the diameter of the silicon single crystal reaches a predetermined value after the formation of the cone section.

The suspension step S4 which follows the straight-body section growing step S3 includes a step of reducing the lifting speed of the silicon single crystal 2 to 0 mm/minute in the state where the upward and downward movement of the crucible 30 is suspended.

The first static-state maintaining step S5 which follows the suspension step S4 includes a step of maintaining the static state where the lifting speed of the silicon single crystal 2 is kept at 0 mm/minute until there is a decrease in the apparent weight of the silicon single crystal 2 measured by the weight measuring unit 5.

The second static-state maintaining step S6 which follows the first static-state maintaining step S5 includes a step of continuing to maintain the static state so that the whole growth front of the silicon single crystal 2 faced with the surface of the melt 32 forms a convex shape which protrudes in the opposite direction to the lifting direction of the silicon single crystal 2 is pulled (hereinafter referred to as "downward convex shape" for simplification).

The suspension step S4, the first static-state maintaining step S5, and the second static-state maintaining step S6 are executed under the influence of the horizontal magnetic field applied to the melt 32 having a magnetic flux density equal to or more than 1000 Gauss, and equal to or less than 2000 Gauss, like the straight-body section growing step S3.

Apart from the present embodiment, the suspension step S4, the first static-state maintaining step S5, and the second static-state maintaining step S6 can also include a step of cutting off the horizontal magnetic field, but this may be attributed to a current change in the melt 32, and it may cause the dislocations.

In the present embodiment, the suspension step S4, the first static-state maintaining step S5, and the second static-state maintaining step S6 should preferably involve continued application of the horizontal magnetic field in order to prevent the dislocations,

accordingly.

The suspension step S4, the first static-state maintaining step S5, and the second static-state maintaining step S6 also involve axial rotations of the silicon single crystal 2 and the crucible 30 in the opposite directions to each other.

The separating step S7, which follows the second static-state maintaining step S6, includes separating the silicon single crystal 2 from the melt 32 by suspending the rewinding of the wire 3 and lifting down the crucible 30.

Referring to Fig. 3, the shape changes of the growth front 7 of the silicon single crystal 2 from the straight-body section growing step S3 to the separating step S7 are described below.

As shown in Fig. 3 (A), the growth front 7 of the silicon single crystal 2 forms a convex shape which protrudes in the direction parallel to the lifting direction of the silicon single crystal (hereinafter referred to as "upward convex shape" for simplification) in the straight-body section growing step S3.

As shown in Fig. 3 (B), the growth front 7 starts to grow in the opposite direction to the lifting direction of the silicon single crystal when the lifting speed of the silicon single crystal 2 reaches 0 mm/minute in the suspension step S4.

As shown in Fig. 3 (B) and Fig. 3 (C), the upward convex shape of the growth front 7 is gradually reduced to become substantially flat, i.e. the growth front 7 is substantially aligned with the fluid surface 9 of the melt 32 in the first static-state maintaining step S5. The growth front 7 then transforms into a downward convex shape, and ends up being located under the fluid surface 9.

The silicon single crystal 2 is free from the buoyancy of the melt 32 and its apparent weight gradually increases with the reduction of the upward convex shape of the growth front 7 due to the growth of the silicon single crystal 2 as long as the growth front 7 is located above the fluid surface 9. However, it is assumed that the silicon single crystal 2 is given the buoyancy of the melt 2 and its apparent weight starts to decrease when the growth front 7 transforms into a downward convex shape to be located under the fluid surface 9.

As shown in Fig. 3 (D), the whole growth front 7 transforms into a downward convex shape due to the maintained static state in the

second static-state maintaining step S6.

Under the influence of the horizontal magnetic field, the whole growth front is more difficult to transform into the downward convex shape and therefore the silicon single crystal 2 is easier to be separated from the melt 32 before the transformation of the whole growing front into the downward convex shape, compared to the absence of the horizontal magnetic field. It is therefore important to bring the whole growth front 7 into the downward convex shape in the first static-state maintaining step S5 and the second static-state maintaining step S6.

The following is a description of Example 1 and 2 as well as Comparative Examples 1-4 of the actual production of silicon single crystals according to the above-described silicon single crystal production method.

In Examples 1, 2 and Comparative Examples 1-4, 240 kg of the raw material was injected into the crucible 30 made of synthetic quartz glass, and the inner pressure of the chamber 20 was reduced to 70 mbar.

In each of Examples 1, 2 and Comparative Examples 1-4, the silicon single crystal 2 was produced under the influence of the horizontal magnetic field applied to the melt 32 having a different magnetic density from one another. The diameter of the produced silicon single crystal 2 was 450 mm.

The magnetic flux density was set at 3000 Gauss in Comparative Examples 1 and 2, 3800 Gauss in Comparative Example 3, and 500 Gauss in Comparative Example 4.

On the other hand, the magnetic flux density was set at 1000 Gauss in Example 1, and 2000 Gauss in Example 2.

Table 1 shows the results from Examples 1, 2 and Comparative Examples 1-4.

Table 1

	weight of raw material (kg)	crucible	inner pressure (mbar)	magnetic flux density (gauss)	results
Comp. Example 1	240	synth. quartz glass	70	3000	the deformation or dislocations in the straight-body section occurred.
Comp. Example 2	240	synth. quartz glass	70	3000	the omission of the tail section was not successful.
Comp. Example 3	240	synth. quartz glass	70	3800	the deformation or dislocations in the straight-body section occurred.
Comp. Example 4	240	synth. quartz glass	70	500	the deformation or dislocations in the straight-body section occurred.
Example 1	240	synth. quartz glass	70	1000	the deformation and dislocations in the straight-body section was suppressed, and the omission of the tail section was successful.
Example 2	240	synthetic quartz glass	70	2000	the deformation and dislocations in the straight-body section was suppressed, and the omission of the tail section was successful.

As shown in Table 1, the generation of the straight-body section was successful in Examples 1 and 2 with the magnetic flux density being equal to or more than 1000 Gauss, and equal to or less than 2000 Gauss.

- 5 On the other hand, the deformation and the dislocations in the straight-body section occurred in Comparative Example 1, 3 and 4 with the magnetic flux density being less than 1000 Gauss, and more than 2000 Gauss.

10 Accordingly, it is understood that the magnetic flux density should be preferably equal to or more than 1000 Gauss, and equal to or less than 2000 Gauss, and this flux density range can achieve the suppression of the deformation and dislocations in the straight-body section.

- 15 On the other hand, it appears that a magnetic flux density less than 1000 Gauss tends to cause the deformation of the silicon single crystals 2; and a magnetic flux density more than 2000 Gauss tends to prevent the growth front 7 from transforming into the downward convex shape.

20 In Comparative Example 2, the omission of the tail section was not successful while the deformation and dislocations in the straight-body section were suppressed.

On the other hand, the omission of the tail section and the suppression of the dislocations in the straight-body section
25 were both successful in Examples 1 and 2.

In the examples which resulted in the failed omission of the tail section, the growth front 7 admittedly transformed into the downward convex shape to some extent as shown in Fig. 4, but the separation of the silicon single crystal 2 from the melt
30 32 still had to take place before the adequate transformation of the whole growth front 7 as the downward convex shape in this example was formed by the partial growth front 7 but not the whole growth front 7.

On the other hand, in the examples which resulted in the
35 successful omission of the tail section, the whole growth front 7 transformed into the downward convex shape as shown in Fig.

5, and therefore the occurrence of the dislocations in the straight-body section was properly suppressed during the separation of the silicon single crystal 2 from the melt 32, even under the influence of the horizontal magnetic field.

5 The following is a description of the effects exerted by the present embodiment.

The present embodiment can achieve the controlled natural current in the melt 32 by lifting up the silicon single crystal 2 under the influence of the horizontal magnetic field with a magnetic flux density being equal to or more than 1000 Gauss,
10 and equal to or less than 2000 Gauss, thereby allowing for the suppression of the deformation and dislocations in the straight-body section 4.

The present embodiment can also prevent the silicon single crystal 2 from being separated from the melt 32 before the adequate transformation of the growth front 7 into the downward convex shape, by suspending the lifting movement of the silicon single crystal 2 relative to the liquid surface of the melt 32 until there is a decrease in the apparent weight of the silicon
15 single crystal 2, and maintaining the static state afterwards so that the whole growth front 7 of the silicon single crystal 2 will form the downward convex shape, thereby allowing for the suppression of the dislocations even under the influence of the horizontal magnetic field and consequently the successful
20 omission of the tail section.

The present invention is not limited to the embodiment described above, and therefore it can be modified within the scope of the appended claim.

For example, unlike in the above embodiment, the driving unit
30 10 can move up/down not only the silicon single crystal 2 but also the crucible 30. In this example, the crucible driver 14 can also rotate or move up/down the crucible 30. The driving unit can also lift up the crucible 30 at the same lifting speed as that of the silicon single crystal 2 so that the relative
35 speed of silicon single crystal 2 to the fluid surface of the melt 32 will be reduced to zero, and then suspend the relative

movement of the silicon single crystal 2 to the melt 32 for a certain period of time.

CLAIM

1. A production method of a silicon single crystal based on the Czochralski method, comprising steps of:

(1) growing a straight body section of said silicon single crystal by lifting up said silicon single crystal;

(2) reducing the relative lifting speed of said silicon single crystal to the surface of said melt to 0 mm/minute, after said step (1);

(3) measuring the apparent weight of the silicon single crystal and maintaining the static state where the relative lifting speed of said silicon single crystal is kept at 0 mm/minute until there is a decrease in the apparent weight of said silicon single crystal, after said step (2);

(4) further maintaining said static state so that the whole growth front of said silicon single crystal faced with said melt will form a convex shape protruding in the opposite direction to the lifting direction of said silicon single crystal, after said step (3), wherein a horizontal magnetic field is applied to a raw material melt contained in a crucible at least during steps (1) to (4), the horizontal magnetic field having a magnetic flux density at its magnetic center which is equal to or more than 1000 Gauss, and equal to or less than 2000 Gauss; and

(5) separating said silicon single crystal from said melt, after said step (4).