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METHOD FOR PRODUCING SILICON INGOTS

The contents of German patent application DE 10 2012 201 735.6 and of German patent application DE 10 2012 203 706.3 is incorporated by reference.
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The invention relates to a method for producing silicon ingots. The invention also relates to a method for producing seed bases for producing a silicon ingot. The invention further relates to seed crystals of crystalline silicon and the use of such seed crystals as a seed base when producing a silicon ingot. Finally the invention relates to a crucible for producing silicon ingots.
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The production of silicon ingots is a fundamental step in the production of silicon wafers, particularly for photovoltaic applications. A method for producing silicon ingots is for instance disclosed in US 2010/0203350 A1. There is always the need to improve methods of this type.
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It is therefore an object of the invention to improve a method for producing silicon ingots. This object is achieved by a method for the production of silicon ingots, the method comprising the following steps:
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- a. providing a vessel for receiving a silicon melt, the vessel comprising
 - i. a bottom which extends in a direction perpendicular to a longitudinal direction;
 - 25 ii. at least one side wall;
- b. arranging a seed structure comprising a plurality of seed bases at the bottom of the vessel,
 - i. the seed bases having a crystal structure with a $\langle 110 \rangle$ direction,



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c. with at least two seed bases being arranged in such a way as to have $\langle 110 \rangle$ directions which are tilted relative to each other at an angle in the range of 0.2° to 10° .

5 The gist of the invention is to arrange seed bases with a known $\langle 110 \rangle$ direction at the bottom of a crucible in such a way that the $\langle 110 \rangle$ directions are tilted relative to each other. They are in particular arranged in such a way that at least two seed bases have $\langle 110 \rangle$ directions which are tilted relative to each other at an angle θ in the range of 0.2° to 10° , particularly in
10 the range of 0.5° to 10° , particularly in the range of 1° to 5° , particularly of less than 3° . It is in particular envisaged to arrange adjacent seed bases correspondingly. Adjacent seed bases in particular have $\langle 110 \rangle$ directions which are tilted relative to each other at an angle θ in the range of 0.2° to 10° , particularly in the range of 0.5° to 10° , particularly in the range of 1°
15 to 5° , particularly of less than 3° . The seed bases are in particular arranged in such a way that the $\langle 110 \rangle$ directions of adjacent seed bases are inclined towards each other. It turned out that using seed bases of this type allows dislocations, occurring in the region of the gaps across the entire block height in a direction parallel to the growth direction, to be reduced. Arrang-
20 ing the seed bases according to the invention thus allows the dislocation density in the silicon ingot to be reduced considerably. This greatly improves the crystal structure of the silicon ingot.

The entirety of the seed bases is part of a seed structure. The seed structure
25 may be disconnected, thus comprising a plurality of separate seed bases which in each case have a particular crystal structure, with the seed bases being separate from each other in the region of the gaps. On the other hand, the seed structure may also be formed as a connected crystal, thus compris-

ing a plurality of seed bases with in each case a particular crystal structure; the gaps between the seed bases, however, have crystallized.

5 Preferably a seed base is arranged in such a way that its $\langle 110 \rangle$ direction is parallel to the longitudinal direction of the crucible. Starting from this seed base, its adjacent seed bases are preferably arranged in such a way that in-
creasingly larger angles are formed between their $\langle 110 \rangle$ directions and the longitudinal direction. This ensures that starting from the bottom of the crucible, the $\langle 110 \rangle$ directions of adjacent seed bases are oriented towards
10 each other.

Preferably the seed bases are symmetrical with respect to a plane which is perpendicular to the bottom of the crucible.

15 Particularly, adjacent seed bases are arranged in such a way that at least one of them has a $\langle 110 \rangle$ direction which is tilted towards a plane that is defined by the respective common gap, is essentially parallel to said gap and is perpendicular to the bottom wall.

20 When providing a silicon melt in the crucible, for instance by melting solid silicon or by pouring in an already molten silicon melt, it is in particular ensured that the seed bases are not completely but in particular only partial-
ly melted. This allows the particular crystal structure and orientation of the seed bases in the silicon melt to be maintained in a subsequent solidifica-
25 tion process.

Preferably the seed bases have dimensions and/or an arrangement at the bottom of the crucible in such a way that the gaps between adjacent seed bases in each case extend in a region in which the finished silicon ingot is

to be cut up. In other words, the seed bases are dimensioned and arranged in such a way that the position of the gaps in the silicon ingot coincides with the position of the saw cuts made during boule production. Correspondingly, it is advantageously intended for the seed bases to be arranged
5 at the bottom of the crucible in such a way that the cuts of the side-slabs coincide with the position of the outermost seed edges. This ensures that the sawing losses due to the sawing process are essentially limited to those regions where, as a result of the gaps, the silicon material exhibits a high dislocation density so that the loss of high-quality silicon material is re-
10 duced.

One way of producing the $\langle 110 \rangle$ direction tilt of a seed is to produce and/or prepare seed bases with an identical geometry in such a way that their $\langle 110 \rangle$ orientations are tilted to various degrees. Alternatively it can
15 be envisaged to use seed bases with a different geometry or to place the seed bases on the bottom of the crucible at an angle thereto. A combination of the three possibilities is conceivable as well.

The seed bases are preferably monocrystalline, i.e. they consist of a single
20 crystal. Preferably they contain no grain boundaries. A so-called quasi-monocrystalline structure of the seed bases, in other words a structure containing large-volume monocrystalline regions and a low number of grain boundaries, is conceivable as well.

25 Another object of the invention is to provide a method for producing seed bases for the production of silicon ingots. This object is achieved by a method for the production of seed bases, the method comprising the following method steps:

- a. providing a monocrystalline silicon block having a known crystal structure;
 - b. dividing the silicon block along a cutting plane,
 - c. with the cutting plane having a normal which forms an angle with a
- 5 $\langle 110 \rangle$ direction of the crystal structure of the silicon block in the range of 0.2° to 10° .

The gist of the invention is to divide a silicon block along a cutting plane, the cutting plane having a normal that forms an angle θ with a $\langle 110 \rangle$ direc-

10 tion of the crystal structure of the silicon block in the range of 0.2° to 10° , in particular in the range of 0.5° to 10° , in particular in the range of 1° to 5° , in particular of less than 3° .

The silicon block to be divided is preferably a single-crystalline or mono-

15 crystalline silicon block.

Another object of the invention is to improve a seed crystal of crystalline silicon, in particular for use as a seed base in the production of a silicon ingot from a silicon melt. These objects are achieved by a seed crystal of

20 crystalline silicon comprising at least one region having

- a. a plane first side which is oriented perpendicular to a first normal; and
- b. a crystal structure having a $\langle 110 \rangle$ direction,
- c. with the $\langle 110 \rangle$ direction forming an angle with the normal in the range of 0.2° to 10° ,

25 and a use of a seed crystal according to the invention as a seed for the production of a silicon ingot from a silicon melt.

The gist of the invention is to produce a seed crystal having a plane first side which is perpendicular to a first normal and a crystal structure with a

<110> direction, the <110> direction forming an angle β with the normal in the range of 0.5° to 10° , in particular in the range of 1° to 5° , in particular of less than 3° . The first side whose normal is tilted relative to the <110> direction may be the upper side or the lower side of the seed crystal.

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The seed crystal preferably has a monocrystalline structure.

The seed crystal may be cuboid-shaped, cylindrical, wedge-shaped or in the shape of a truncated wedge.

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The seed crystal can in particular be used as a seed base in the production of a silicon ingot from a silicon melt.

Another object of the invention is to improve a crucible for a method for
15 producing silicon ingots. This object is achieved by a crucible for a method according to the invention, comprising a longitudinal direction, at least one side wall and a bottom, wherein the bottom is, starting from a central region, formed in such a way as to rise towards the side wall and the surface normal of various bottom regions form a tilting angle of 0.5° to 10° relative
20 to each other. The advantages are the same as described above.

The crucible in particular has a bottom which, on a side facing the interior of the crucible, is provided with several regions which are in each case plane and have a surface normal, with the surface normals of at least two
25 regions, in particular adjacent regions, being tilted relative to each other.

In other words, the bottom plate of the crucible is concave on its side facing the interior, i.e. the interior forms a convex polyhedron which is open on one side.

Further features and details of the invention will become apparent from the description of several embodiments with reference to the drawings in which

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Fig. 1 shows a diagrammatic cross-section through a crucible for producing silicon ingots, the bottom of which being provided with an arrangement of seed bases;

10 Fig. 2 shows a view similar to Fig. 1 with an alternative arrangement of the seed bases;

Fig. 3 shows an example illustrating the effects of the various arrangements of the seed bases at the bottom of the crucible; and

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Fig. 4 shows a view of a crucible according to the invention.

In a method for producing silicon ingots 1, a vessel 2 designed as a crucible or coquille for receiving a silicon melt 3 is provided in a first step.

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The vessel 2 has a bottom 5, which is perpendicular to a longitudinal direction 4, and four side walls 6 which have at least a component extending in the longitudinal direction. The bottom 5 is in the shape of a square, i.e. the vessel 2 has a square cross-section. It is however conceivable as well for the vessel 2 to have a different cross-section, in particular a round, in particular a circular cross-section.

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At the bottom 5 of the vessel 2, there is arranged a plurality of seed bases 7. The entirety of the seed bases 7 is referred to as seed structure. The seed

bases 7 are preferably of a monocrystalline silicon crystal. They have in particular no grain boundaries. They generally have a crystal structure with a known $\langle 110 \rangle$ direction 8 which is shown in Figs. 1 and 2 for illustration purposes. The seed bases 7 are in each case separated from each other by gaps 9 between the seeds. The gaps 9 between the seeds are in particular linear.

In a direction perpendicular to the longitudinal direction 4, the seed bases 7 have a rectangular, particularly a square cross-section. They are in particular in the shape of a cuboid. They have in particular one plane first side 10 each which is in each case perpendicular to a first normal 11. The first side 10 is in particular the lower side of the seed bases 7. The first side 10 may however also be the upper side of the seed bases 7.

The seed bases 7 are arranged at the bottom 5 of the vessel 2 in such a way that at least two different seed bases 7 have different $\langle 110 \rangle$ directions 8. At least two seed bases 7 are arranged in such a way as to have $\langle 110 \rangle$ directions 8 which are tilted relative to each other at an angle b in the range of 0.2° to 10° , in particular in the range of 0.5° to 10° , in particular in the range of 1° to 5° , in particular of no more than 3° . Particularly it is intended for adjacent seed bases 7 to be in each case arranged in such a way that their $\langle 110 \rangle$ directions 8 are in each case tilted relative to each other at an angle b in the range of 0.2° to 10° , in particular in the range of 0.5° to 10° , in particular in the range of 1° to 5° , in particular of less than 3° . The seed bases 7 are tilted in such a way that their $\langle 110 \rangle$ directions 8 are tilted towards each other in the longitudinal direction 4, i.e. starting from the bottom 5 of the vessel 2.

One of the seed bases 7, in particular a central seed base 7, may preferably be arranged in such a way that its $\langle 110 \rangle$ direction 8 is parallel to the longitudinal direction 4.

- 5 Starting from the central seed base 7, the seed bases 7 are arranged in such a way that increasingly larger angles b_i are formed between their $\langle 110 \rangle$ directions 8 and the longitudinal direction 4 which is shown as a dashed arrow in each of the Figures 1 and 2. The closer a seed base 7 is arranged to the edge, i.e. the closer to the side wall 6, the larger the angle b_i formed
- 10 with the longitudinal direction 4. This ensures that all seed bases 7 have $\langle 110 \rangle$ directions 8 such that the $\langle 110 \rangle$ directions 8 of adjacent seed bases 7 are in each case tilted towards each other. Particularly, adjacent seed bases 7 are arranged in such a way that at least one of them has a $\langle 110 \rangle$ direction 8 which is tilted towards a plane which is defined by the respective
- 15 gap 9 between them and is perpendicular to the bottom 5.

- Except for the gaps 9 between the seeds and – if present – an edge gap 12, preferably the entire bottom surface of the vessel 2 is covered with seed bases 7. There are in particular provided 5 x 5 seed bases 7 having a square
- 20 cross-section. It is however conceivable as well to use 1 x 5 seed bases 7 having a rectangular, striped cross-section. In this case, the longer side of the seed base 7 preferably just corresponds to the extension of the bottom 5 in a direction perpendicular to the longitudinal direction 4 parallel to the side wall 6.

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The seed bases 7 are in particular mirror-symmetric to a central plane which is perpendicular to the bottom 5 of the vessel 2.

In the direction perpendicular to the longitudinal direction 4, the seed bases 7 in particular have a width B which just corresponds to an integral multiple of a width of boules to be sawed out of the silicon ingot 1; if necessary, the width is chosen such as to include the respective sawing gaps. The
5 width B of the seed bases 7 may in particular essentially just be equal to the final boule width. This means that it differs from the width of the boules to be sawed out of the silicon ingot 1 by no more than 10%, in particular by no more than 5%. Correspondingly, the edge gaps 12 may have dimensions in the direction perpendicular to the longitudinal direction 4 which corre-
10 spond to a thickness of the flanks to be removed.

Starting from the central seed base 7 of the embodiment shown in Fig. 1, the $\langle 110 \rangle$ direction 8 of the seed bases 7 forms increasingly larger angles b_i with the first normal 11. This can be ensured by a particular way of pro-
15 ducing or preparing the seed bases 7 which will be explained in more detail below.

Alternatively, it is conceivable as well to place the seed bases 7 on the bottom 5 of the vessel 2 at an angle thereto, as shown in Fig. 2. In this embod-
20 iment, the $\langle 110 \rangle$ direction 8 of each seed base 7 may just coincide with its respective first normal 11.

In order to precisely define the position of the seed bases 7 on the bottom 5 of the vessel 2, particularly in order to precisely define the $\langle 110 \rangle$ direction
25 tilt relative to the longitudinal direction 4 of the vessel 2, spacers 15 are provided at the bottom 5 of the vessel 2. The spacers 15 may in particular be wedge-shaped or in the shape of a truncated wedge. Alternatively, it is conceivable as well to structure the bottom 5 of the vessel 2 in such a way

that the $\langle 110 \rangle$ direction 8 of the seed bases 7 is in each case tilted relative to the longitudinal direction 4 of the vessel 2 as required.

After arranging the seed bases 7 at the bottom 5 of the vessel 2, the silicon melt 3 is provided in the vessel 2. To this end, solid silicon may be arranged and melted in the vessel 2. It is conceivable as well to melt silicon in a separate vessel and to fill it into the vessel 2 in liquid form, i.e. as silicon melt 3.

10 In both alternatives, a corresponding temperature control ensures that the seed bases 7 will melt only partially but not completely. When seen in the longitudinal direction 4, no more than 70%, in particular no more than 50%, in particular no more than 30% of the seed bases 7 are melted.

15 Afterwards, the silicon melt 3 is solidified in a targeted manner. Details concerning the melting of silicon and solidifying the silicon melt 3 can be found in DE 10 2005 013 410 B4.

Once the silicon melt 3 has solidified to form the silicon ingot 1, the silicon ingot 1 is sawed into boules with cuts that are parallel to the longitudinal direction 4. In this process, flanks, which are extensions of the edge gaps 12, occur as a waste product. The silicon ingot 1 is in particular sawed into pieces by cutting it in a direction parallel to the longitudinal direction 4 so that the sawing cuts are a straight extension of the longitudinal direction 4 of a gap 9 between the seeds. This reduces the cutting loss of high-quality silicon.

Furthermore, a bottom and a cap of the silicon ingot 1 are removed by cutting in a direction perpendicular to the longitudinal direction 4. The bot-

tom, which contains the original seed bases 7 with the gaps 9 between them filled by crystallization may advantageously be used as a seed structure for a subsequent crystallization process.

- 5 Fig. 3 shows an exemplary cross-section through a silicon ingot 1 produced in a manner according to the invention. The seed bases 7 corresponding to the individual regions and their respective $\langle 110 \rangle$ directions 8 are shown for illustration purposes as well. As can be seen from Fig. 3, arranging the seed bases 7 with their $\langle 110 \rangle$ directions 8 tilted towards each other consid-
10 erably reduces the extension of dislocation zones 13.

The following is a description of a method for producing the seed bases 7. In order to produce the seed bases 7, a monocrystalline silicon block having a known crystal structure is provided and divided along cutting planes.

- 15 The cutting planes have in each case one normal which forms an angle with a $\langle 110 \rangle$ direction of the crystal structure of the silicon block in the range of 0.2° to 10° , in particular in the range of 0.5° to 10° , in particular in the range of 1° to 5° , in particular of no more than 3° . The seed bases 7 may be produced from the monocrystalline silicon block by means of two such
20 cutting planes which are spaced from each other. The cutting planes may in each case be parallel to each other. Also, they can each have normals which are tilted relative to each other at an angle in the range of 0.2° to 10° , in particular in the range of 0.5° to 10° , in particular in the range of 1° to 5° , in particular of no more than 3° . The first alternative is particularly advan-
25 tageous for producing cuboid seed bases 7. The second alternative allows wedge shaped seed bases 7 or seed bases 7 in the shape of a truncated wedge to be produced.

Generally speaking these seed bases 7 are block regions, particularly cuboids made of silicon, particularly monocrystalline silicon, whose crystal structure is oriented in such a way that one of their $\langle 110 \rangle$ directions forms an angle with the first normal 11 on one of their plane sides 10 in the range of 0.2° to 10° , in particular in the range of 0.5° to 10° , in particular in the range of 1° to 5° , in particular of no more than 3° .

Generally speaking, the seed crystals are cuboid-shaped, cylindrical, wedge-shaped or in the shape of a truncated wedge. They can in particular be used as seed bases 7 in the production of the silicon ingot 1 from the silicon melt 3.

According to another aspect of the invention, the seed bases 7 are tilted by a particular design of the bottom 5 of the vessel 2. The vessel 2 in particular has a bottom 5 which is formed in such a way as to rise from a central region towards the side wall 6. Different bottom regions have in each case one surface normal which are tilted relative to each other at a tilting angle in the range of 0.2° to 10° , in particular in the range of 0.5° to 10° .

The bottom 5 rises continuously towards the side wall 6. The bottom 5 in particular has several distinct plane regions in which the rise of these regions in the direction of a side wall 6 increases.

In other words, on its side facing the interior of the vessel 2, the bottom 5 comprises several regions which are in each case plane and have in each case surface normals 11, with the surface normals 11 of at least two regions being tilted relative to each other at an angle in the range of 0.2° to 10° , in particular in the range of 0.5° to 10° . In particular the surface normals 11 of

two adjacent regions are in each case tilted relative to each other. Therefore, the bottom wall 5 has a concave shape on its side facing the interior.

Claims

1. A method for the production of silicon ingots (1), the method comprising the following steps:
 - 5 a. providing a vessel (2) for receiving a silicon melt (1), the vessel (2) comprising
 - i. a bottom (5) which extends in a direction perpendicular to a longitudinal direction (4);
 - ii. at least one side wall (6);
 - 10 b. arranging a seed structure comprising a plurality of seed bases (7) at the bottom (5) of the vessel (2),
 - i. the seed bases (7) having a crystal structure with a $\langle 110 \rangle$ direction,
 - c. with at least two seed bases (7) being arranged in such a way as to
 - 15 have $\langle 110 \rangle$ directions (8) which are tilted relative to each other at an angle (a) in the range of 0.2° to 10° .
2. A method according to claim 1, **characterized in that** a seed (7) is arranged in such a way that its $\langle 110 \rangle$ direction (8) is aligned parallel
20 with the longitudinal direction (4).
3. A method according to claim 2, **characterized in that** starting from a first seed (7), adjacent seed bases (7) are arranged in such a way that their $\langle 110 \rangle$ directions (8) are tilted relative to the $\langle 110 \rangle$ direction (8)
25 of the first seed (7) at increasingly larger angles.
4. A method according to claim 1, **characterized in that** one seed (7) is arranged in such a way as to have a $\langle 110 \rangle$ direction (8) which is tilted towards the longitudinal direction (4).

5. A method according to claim 1, **characterized in that** in at least one of the seed bases (7), the $\langle 110 \rangle$ direction (8) forms an angle (b) with a normal (11) to a plane side (10) of the corresponding seed (7) in the range of 0.2° to 10° .
6. A method according to claim 1, **characterized in that** a normal (11) to a plane side (10) of at least one of the seed bases (7) forms an angle with the longitudinal direction (4) of the vessel (2) in the range of 0.2° to 10° .
7. A method according to claim 1, **characterized in that** in at least one of the seed bases (7), the $\langle 110 \rangle$ direction (8) forms an angle (b) with the longitudinal direction (4) in the range of 0.2° to 10° .
8. A method for the production of seed bases (7), the method comprising the following method steps:
- providing a monocrystalline silicon block (1) having a known crystal structure;
 - dividing the silicon block (1) along a cutting plane,
 - with the cutting plane having a normal which forms an angle (b) with a $\langle 110 \rangle$ direction of the crystal structure of the silicon block (1) in the range of 0.2° to 10° .
9. A seed crystal of crystalline silicon comprising at least one region having
- a plane first side (10) which is oriented perpendicular to a first normal (11); and
 - a crystal structure having a $\langle 110 \rangle$ direction (8),

- c. with the $\langle 110 \rangle$ direction (8) forming an angle (b) with the normal (11) in the range of 0.2° to 10° .

10. A seed crystal according to claim 9, **characterized in that** said seed
5 crystal has several regions with crystal structures whose $\langle 110 \rangle$ directions (8) are tilted relative to each other at an angle (a) in the range of 0.2° to 10° .
11. A use of a seed crystal according to claim 9 as a seed (7) for the pro-
10 duction of a silicon ingot (1) from a silicon melt (3).
12. A crucible (2) for a method according to claim 1, comprising a longi-
tudinal direction (4), at least one side wall (6) and a bottom (5), where-
in the bottom (5) is, starting from a central region, formed in such a
15 way as to rise towards the side wall (6) and the surface normal of vari-
ous bottom regions form a tilting angle of 0.5° to 10° relative to each
other.
13. A crucible (2) according to claim 12, wherein the bottom (5) rises con-
20 tinuously towards a side wall (6).
14. A crucible (2) according to claim 12, wherein the bottom (5) comprises
several distinct plane regions in which the rise of these regions in the
direction of a side wall (6) increases.