GROWTH OF PLURAL SAMPLE RODS TO DETERMINE IMPURITY BUILD-UP DURING PRODUCTION OF SINGLE CRYSTAL SILICON INGOTS

~~FIELD OF THE DISCLOSURE~~CROSS-REFERENCE TO RELATED APPLICATION

[0001] This application claims priority to U.S. Patent Application No. 16/020,701 filed on 27 June 2018, the entire disclosure of which is hereby incorporated by reference in its entirety.

FIELD OF THE DISCLOSURE

[0002] The field of the disclosure relates to methods for forming single crystal silicon ingots in which plural sample rods are grown from the melt. A parameter related to the impurity concentration(s) of the melt or ingot is measured. In some embodiments, the sample rods each have a diameter less than the diameter of the product ingot.

BACKGROUND

[0003] Single crystal silicon, which is the starting material for most processes for the fabrication of semiconductor electronic components, is commonly prepared by the so-called Czochralski (CZ) process wherein a single seed crystal is immersed into molten silicon and then grown by slow extraction. Molten silicon is contaminated with various impurities, among which is mainly oxygen, during the time it is contained in a quartz crucible. Some applications, such as advanced wireless communication applications, insulated gate bipolar transistors (IGBT) and low power, low leakage devices, require wafers with a relatively high resistivity such as 1500 ohm-cm (Ω-cm) or more.

[0030] In accordance with embodiments of the present disclosure, after polycrystalline silicon is added to the crucible 22 and the heating system 39 is operated to melt-down the polycrystalline silicon, plural sample ingots or rods are pulled from the melt. Generally, the plural sample rods have a diameter smaller than the subsequently grown product ingot (e.g., less than about 150 mm, less than about 100 mm, less than about 50 mm, less than about 25 mm). An example sample rod 5 is shown in Figure 2. The rod 5 includes a crown 21 in which the rod transitions and tapers outward from the seed to reach a target diameter. The rod 5 includes a constant diameter portion 25 or cylindrical main body or simply "body", of the crystal which is grown by increasing the pull rate. The main body 25 of the sample rod 5 has a relatively constant diameter. The rod 5 includes a tail or end-cone 29 in which the rod tapers in diameter after the main body 25. When the diameter becomes small enough, the rod is then separated from the melt. The rod 5 has a central longitudinal axis A that extends through the crown 21 and a terminal end 33 of the ingot.

[0031] After the first sample rod is pulled from the melt, a first sample rod parameter related to the quality of the first sample rod and/or the silicon melt is measured. The measured parameter may be related to the impurity make-up of the rod such as the phosphorous concentration, boron concentration, total impurity concentration, resistivity of the sample rod, or other impurity concentration (e.g., aluminum, gallium, arsenic, indium, antimony, and the like).

is caused to contact the rod 5 on the flat planar segment of the rod. Current is passed through the supports 53, 55 and the voltage is measured by the probe tip 61. The probe tip 61 is moved manually down the axis of the neck 5 with current applied/voltage being measured at each point. In the illustrated apparatus 43 the probe tip 61 is moved manually. In other embodiments, the probe tip 61 is moved by actuators.

[0045] The measurement apparatus 43 of Figure 4 is an example apparatus and any suitable apparatus for securing and/or measuring the resistivity of the rod may be used unless stated otherwise. Use of a rod (e.g., generally ~~s~~narrow diameter rod such as less than 100 mm, 50 mm or less than 25 mm) and the measurement apparatus 43 allows the resistivity to be measured without slicing the rod into wafers or slugs.

[0046] The resistivity probe may be a two point probe in which both probe tips are contacted with the planar segment 11. The voltage difference is measured across the two probe tips. For example, resistivity may be measured with a two-point probe in accordance with SEMI Standard MF397-0812, entitled "Test Method for Resistivity of Silicon Bars using a Two-Point Probe," which is incorporated herein by reference for all relevant and consistent purposes. The 2-terminal or 3-term~~a~~inal rectification method may be used to determine the crystal type (i.e., N-type or P-type). Such type determination may be performed in accordance with SEMI Standard MF42-0316, entitled "Test Method for Conductivity Type of Extrinsic Semiconducting Materials", which is incorporated herein by reference for all relevant and consistent purposes. Both

the oxygen content of each sample rod is less than 5.2 ppma, less than 5.0 ppma, less than 3.5 ppma, less than about 3 ppma or even less than about 2.5 ppma. In some embodiments, the sample rod 5 is free of dislocations.

[0051] In some embodiments, a product ingot is grown from the melt after the sample rods are grown. The diameter of the first sample rod and the diameter of the second sample rod are less than the diameter of the product ingot. For example, the diameter of the sample rods may be less than 0.75 times ~~the times~~ the diameter of the product ingot, less than 0.50 times, less than about 0.25 times or less than 0.1 times the diameter of the product ingot.

[0052] In some embodiments, the rod 5 has a diameter that generally corresponds to the diameter of the neck portion of a product ingot grown in the crystal puller. For example, the rod may have a diameter of less than 50 mm, less than 25 mm, or less than 20 mm.

[0053] In some embodiments, polycrystalline silicon is not added during the growth of the product ingot (e.g., as in a batch process). Additional sample rods may be grown after the product ingot is grown and the crucible is recharged with polycrystalline silicon. In other embodiments, polycrystalline silicon is added to the melt as the product ingot is grown (e.g., as in a continuous Czochralski method).

[0054] The measured first sample rod parameter and the measured second sample rod parameter may be used to adjust the growth conditions of the product ingot and/or of one or more subsequently grown product ingots pulled from other melts (e.g., a second melt formed in the crucible

~~WHAT IS CLAIMED IS~~CLAIMS:

1. A method for producing a single crystal silicon ingot from a silicon melt held within a crucible comprising:

adding polycrystalline silicon to the crucible;

heating the polycrystalline silicon to cause a silicon melt to form in the crucible;

pulling a first sample rod from the melt, the first sample rod having a first sample rod diameter;

measuring a first sample rod parameter related to the quality of the first sample rod and/or the silicon melt;

pulling a second sample rod from the melt, the second sample rod having a second sample rod diameter, dopant not being added to the melt before growth of the second sample rod;

measuring a second sample rod parameter related to the quality of the second sample rod and/or the silicon melt; and

pulling a product ingot from the melt, the product ingot having a diameter, the first sample rod diameter and the second sample rod diameter being less than the diameter of the product ingot.

2. The method as set forth in claim 1 wherein the first sample rod parameter and the second sample rod parameter are the same parameter.

3. The method as set forth in claim 2 further comprising comparing the measured first sample rod parameter to the measured second sample rod parameter.

4. The method as set forth in claim 3 wherein the change of the parameter over time is measured.

5. The method as set forth in claim 1 wherein the first sample rod parameter and the second sample rod parameter are different parameters.

6. The method as set forth in any one of claims 1 to 5 wherein the first sample rod parameter and the second sample rod parameter is related to the impurity concentration of the melt and/or product ingot.

7. The method as set forth in claim 6 wherein the parameter is selected from the group consisting of the phosphorous concentration, boron concentration, aluminum concentration, gallium concentration, arsenic concentration, indium concentration, antimony concentration, total impurity concentration, dopant concentration, and resistivity of the sample rod.

8. The method as set forth in any one of claims 1 to 7 wherein polycrystalline silicon is not added to the crucible after the first sample rod is grown.

9. The method as set forth in any one of claims 1 to 8 wherein the impurity concentration of the first rod and second rod are measured, the concentration of impurities in the first and second rods being less than $1x10^{12}$ atoms/cm^3.

10. The method as set forth in claim 9 wherein the impurity concentration is measured by Fourier-transform infrared spectroscopy or by photoluminescence.

11. The method as set forth in any one of claims 1 to 10 further comprising altering a growth condition of the product ingot based at least in part on the measured first sample rod parameter and/or the measured second sample rod parameter.

12. The method as set forth in any one of claims 1 to 10 wherein the product ingot is a first product ingot and the melt is a first melt, the method further comprising altering a growth condition of a second product ingot grown from a second melt based at least in part on the measured first sample rod parameter and/or the measured second sample rod parameter.

13. The method as set forth in claim 11 or claim 12 wherein the growth condition is selected from the group consisting of a hot-zone configuration, side heater power, heater geometry, crucible liner thickness, dopant concentration, impurity and dopant evaporation, gas flow rate, and chamber pressure.

14. The method as set forth in any one of claims 1 to 13 further comprising correlating the measured first sample rod parameter and/or the measured second sample rod parameter to crucible dissolution, hot zone contamination or gas flow impurity.

15. The method as set forth in any one of claims 1 to 14 wherein the first sample rod parameter and the second sample rod parameter are the resistivity of the sample rod, the first sample rod resistivity and the second sample rod resistivity being measured by contacting the rod with a resistivity probe.

16. The method as set forth in claim 15 further comprising:

forming a planar segment on the first sample rod, the resistivity of the first sample rod being measured on the planar segment; and

forming a planar segment on the second sample rod, the resistivity of the second sample rod being measured on the planar segment.

17. The method as set forth in claim 16 wherein each planar segment extends axially from one end of the sample rod toward a second end of the sample rod.

18. The method as set forth in any one of claims 15 to 17 further comprising applying a current to the sample rod to measure a resistance of the sample rod.

19. The method as set forth in any one of claims 15 to 18 wherein each sample rod is secured by a measurement apparatus comprising a clamp that holds the sample rod while contacting the sample rod with a resistivity probe.

20. The method as set forth in any one of claims 15 to 19 further comprising annealing each sample rod in a thermal donor kill cycle prior to measuring a resistivity of the sample rod.

21. The method as set forth in any one of claims 1 to 20 wherein the diameters of the first and second sample rods are each ~~less than 0.75 times the diameter of the product ingot, or~~ less than 0.50 times~~, less than about 0.25 times or less than 0.1 times~~ the diameter of the product ingot.

22. The method as set forth in any one of claims 1 to 21 wherein the first and second sample rods each have a length of less than about 300 mm~~, less than about 200 mm or less than about 100 mm~~.

23. The method as set forth in any one of claims 1 to 22 wherein the product ingot has a resistivity of at least about 1,500 Ω-cm~~, or at least about 2,000 Ω-cm, at least about 4,000 Ω-cm, at least about 6,000 Ω-cm, at least about 8,000 Ω-cm, at least about 10,000 Ω-cm, from about 1,500 Ω-cm to about 50,000 ohm-cm or from about 8,000 Ω-cm to about 50,000 Ω-cm~~.

24. The method as set forth in any one of claims 1 to 23 wherein the first and second sample rod each has a resistivity of at least ~~about~~ 1,500 Ω-cm~~, or at least about 2,000 Ω-cm, at least about 4,000 Ω-cm, at least about 6,000 Ω-cm, at least about 8,000 Ω-cm, at least about 10,000 Ω-cm, from about 1,500 Ω-cm to about 50,000 ohm-cm or from about 8,000 Ω-cm to about 50,000 Ω-cm~~.

25. A method for modeling the impurity concentration of a single crystal silicon ingot, the method comprising:

pulling a first sample rod from a silicon melt disposed in a crucible, the first sample rod having a first sample rod diameter of less than 100 mm;

measuring a first sample rod parameter related to the impurity content of the first sample rod;

pulling a second sample rod from the silicon melt, the second sample rod having a first sample rod diameter of less than 100 mm;

measuring a second sample rod parameter related to the impurity content of the second sample rod;

determining the impurity concentration of a single crystal silicon ingot based at least in part on a rate of change in the difference between the measured first sample rod parameter and the measured second sample rod parameter.

26. The method as set forth in claim 25 wherein the first sample rod parameter and the second sample rod parameter is the same parameter, the parameter being selected from the group consisting of the phosphorous concentration, boron concentration, aluminum concentration, gallium concentration, arsenic concentration, indium concentration, antimony concentration, total impurity concentration, dopant concentration, and resistivity of the sample rod.

27. The method as set forth in claim 25 or claim 26 wherein the first sample rod diameter and the second sample rod diameter is each less than about 50 mm~~, less than about 25 mm, less than about 20 mm, from about 5 mm to about 100 mm, from about 5 mm to about 50 mm, from about 5 mm to about 25 mm or from about 10 mm to about 25 mm~~.

28. The method as set forth in any one of claims 25 to 27 further comprising:

pulling a third, fourth, fifth, sixth, seventh, eighth, ninth and/or tenth sample rod from the melt, the diameter of each sample rod being less than about 100 mm; and

measuring a sample rod parameter related to the impurity content for each sample rod pulled from the melt.

~~29. The method as set forth in any one of claims 25 to 28 wherein the impurity concentration of a single crystal silicon ingot is determined from at least in part a rate of change in the difference between the measured first sample rod parameter and the measured second sample rod parameter.~~

~~30~~29. The method as set forth in any one of claims 25 to ~~29~~28 wherein the impurity concentration of a single crystal silicon ingot is determined from a model, the first sample rod parameter and the second sample rod parameter being input into ~~to~~ the model.

~~31~~30. The method as set forth in any one of claims 25 to ~~30~~29 further comprising pulling a product ingot from the melt, the single crystal silicon ingot in which the impurity concentration is determined being the product ingot pulled from the melt.

~~32~~31. The method as set forth in any one of claims 25 to ~~31~~30 wherein the melt is a first melt, the method further comprising:

pulling a first product ingot from the first melt: and

pulling a second product ingot from a second melt, the single crystal silicon ingot in which the impurity concentration is determined being the second product ingot pulled from the second melt.

~~33~~32. The method as set forth in claim ~~31~~30 or claim ~~32~~31 wherein the diameter of the first and second sample rods are each less than 0.75 times the diameter of the product ingot~~, or less than 0.50 times, less than about~~

~~0.25 times or less than 0.1 times the diameter of the product ingot~~.

~~34~~33. The method as set forth in any one of claims 25 to ~~33~~32 wherein the impurity concentration is an impurity concentration of a portion of the single crystal silicon ingot.

~~35~~34. The method as set forth in any one of claims 25 to ~~34~~33 wherein the impurity concentration is concentration of one or more impurities over the length of the single crystal silicon ingot.

~~36~~35. The method as set forth in any one of claims 25 to ~~34~~33 further comprising annealing each sample rod in a thermal donor kill cycle prior to measuring the first and second sample rod parameters.