PURIFICATION OF A METALLOID BY CONSUMABLE ELECTRODE VACUUM ARC REMELT PROCESS

Cross Reference To Related Applications

[0001] This application claims the benefit of U.S. Provisional Application No. 61/527,799 filed August 26, 2011, hereby incorporated herein by reference in its entirety.

Field of the Invention

[0002] The present invention relates to production of a purified metalloid such as silicon by a consumable electrode vacuum arc remelt process.

Background of the Invention

[0003] High purity silicon is needed for many kinds of electronic components such as silicon transistors, silicon integrated circuits and silicon solar cells. Since the invention of the first silicon transistor, many processes have been developed for producing silicon having the required purity levels.

[0004] A process that has been used for producing high quality metals such as steels, nickel-based superalloys, titanium etc. is known as a consumable electrode vacuum arc remelt (CEVAR) process. See, for example, United States Patent Nos. 3,187,079 (Pestel); 3,344,840 (Buehl et al.); 3,480,716 (Lynch et al.); 4,303,797 (Roberts); 4,569,056 (Veil, Jr.); and United States Patent Application Publication No. 2008/0142188 A1 (Ishigami) for various technical aspects of the CEVAR process, all of which publications are incorporated herein in their entireties by reference. The CEVAR process is differentiated from a non-consumable electrode vacuum arc remelt where a non-consumable electrode, for example a graphite or tungsten electrode, is used to melt titanium or zirconium, for example, as disclosed in United States Patent No. 3,546,348 ~~(Serafino)~~. United States Patent Application Publication No. 2010/0154475 A1 (Matheson et al.) discloses a primary silicon purification process with similarities to the Kroll purification process of titanium with brief mention of a secondary silicon composition purification process that involves high temperature vacuum melting of a silicon composition that comprises a boron and phosphorus doped silicon with silicon purity in the range of 99.99 per cent to 99.9999 percent.

[0005] Generally the CEVAR process produces a purified metal by these four steps: (1) evaporating impurities as the metal electrode is melted and exposed to a vacuum in the CEVAR furnace; (2) floating out of the liquid (melted) metal impurities that have a lower density than the

[0019] The CEVAR purification process utilized in the present invention is generally similar to that described, for example, in the above disclosed prior art except for preheat of the silicon electrode used in the CEVAR process and the use of a short CEVAR open-bottomed, water-cooled metal crucible as disclosed herein. Generally for the present invention, during the CEVAR purification process, the preheated silicon electrode is placed in a short CEVAR open-bottomed crucible within the CEVAR furnace that is made gas-tight and brought to a vacuum or an otherwise controlled environment. During the process, direct current (DC current) ~~flows~~ through the electrode and the melt formed below the electrode establishes an arc between the lower end of electrode and the top of the melt with the arc zone remaining within the height of the short CEVAR opened-bottom crucible so that a hot (at a temperature elevated above room temperature) solidified ingot exits the bottom of the short CEVAR opened-bottom crucible. Further controlled cooling of the hot solidified ingot exiting from the short CEVAR opened-bottom crucible is, as further described below, performed to substantially avoid cracking of the solidified ingot.

[0020] The ingot is withdrawn from the short CEVAR crucible into a heater system that provides controlled cooling over the temperature range in which the ingot is likely to crack. The ingot withdrawal rate is essentially matched to the ingot growth rate during steady state operation, so that the arc zone and the top of the ingot remain within the crucible. In an alternative arrangement of the present invention, the ingot is kept stationary and the crucible, electrode and the crucible's exit heater are raised together, to essentially match the growth rate of the ingot.

[0021] In practice of one embodiment of the present invention, the following process steps are performed: (1) forming an electrode from one or more pieces of silicon; (2) preheating the electrode to a temperature (by way of example and not limitation, between 800 and 1200 degrees Centigrade) at which it becomes sufficiently conductive (with a CEVAR process resistivity) to pass current in a subsequent CEVAR processing step without excessive voltage drop and prevention of cracking of the electrode ; (3) melting the electrode by a CEVAR process; (4) causing the resulting hot ingot to pass into a heating system adjacent to the open-bottomed CEVAR crucible while the ingot is at a sufficiently elevated temperature to prevent cracking; and (5) controlling the heating system so as to prevent cracking of the silicon ingot as it cools.

[0022] In alternative examples of the invention, the above preheating process step (2) may be performed within the CEVAR furnace or ~~externally~~ to the furnace as described above.

CLAIMS

1. A method of producing a silicon ingot from a silicon electrode in a CEVAR purification process performed in a short CEVAR open-bottomed crucible disposed in a CEVAR furnace, the method comprising:

heating the silicon electrode to a temperature below the melting point of the silicon electrode prior to initiation of the CEVAR purification process to form a preheated silicon electrode having a CEVAR process resistivity;

melting the preheated silicon electrode by the CEVAR purification process for formation of a silicon ingot at an elevated temperature at the open bottom of the short CEVAR opened-bottomed crucible;

passing the silicon ingot at the elevated temperature through a heating system adjacent to the short CEVAR open-bottomed crucible; and

regulating the heating system to provide a temperature-controlled thermal environment for the silicon ingot at the elevated temperature as the silicon ingot exits the open-bottomed crucible to cool the silicon ingot without cracking.

2. The method of claim 1 where heating the silicon electrode is performed in an external heating chamber prior to placing the preheated silicon electrode in the CEVAR furnace and transfer of the preheated silicon electrode from the external heating chamber to the CEVAR furnace is accomplished in a controlled environment.

3. The method of claim 1 where heating the silicon electrode is performed subsequent to placing the silicon electrode within the CEVAR furnace.

4. The method of claim 1 where heating the silicon electrode further comprises heating the silicon electrode to a temperature within the range of 800 to 1,200 degrees Centigrade to form the preheated silicon electrode.

5. The method of claim 1 further comprising thermally insulating the preheated silicon electrode while melting the preheated silicon electrode by the CEVAR purification process.

6. The method of claim 1 further comprising an auxiliary heater apparatus disposed within the CEVAR furnace to heat the preheated silicon electrode during the CEVAR purification process.

7. A CEVAR furnace system for producing a purified silicon ingot from a silicon electrode, the CEVAR furnace system comprising:

a silicon electrode heating apparatus for preheating the silicon electrode to form a preheated silicon electrode;

a gas-tight CEVAR furnace chamber;

a short CEVAR open-bottomed crucible for containment of an arc zone from a CEVAR purification process melting the preheated silicon electrode, the short CEVAR open-bottomed

crucible disposed in the gas-tight CEVAR furnace chamber;

a preheated silicon electrode drive system for lowering the preheated silicon electrode within the short CEVAR open-bottomed crucible as the lower end of the preheated silicon electrode melts in the CEVAR purification process;

an ingot heating apparatus disposed adjacent to the open bottom of the short CEVAR open-bottomed crucible through which the silicon ingot formed in the CEVAR purification process passes;

an ingot heating controller for controlling the ingot heating apparatus to provide a temperature-controlled thermal environment for the silicon ingot passing through the ingot heating apparatus; and

an ingot withdrawal drive system for alternatively withdrawing the silicon ingot from the short CEVAR open-bottomed crucible at a vertical growth rate of the silicon ingot during steady state of the CEVAR purification process or raising the short CEVAR open-bottomed crucible, the silicon electrode and the ingot heating apparatus at the vertical growth rate of the silicon ingot during steady state of the CEVAR purification process.

8. The CEVAR furnace system of claim 7 wherein the interior height of the short CEVAR opened-bottom crucible is at least 60 percent and less than 120 percent of the diameter of the silicon ingot formed in the CEVAR purification process.

9. The CEVAR furnace system of claim 7 further comprising a vacuum lock chamber connected between the silicon electrode heating apparatus and the short CEVAR opened-bottom crucible in the CEVAR furnace to prevent exposure to air of the preheated silicon electrode during transfer from the silicon electrode heating apparatus to the gas-tight CEVAR furnace chamber.

10. The CEVAR furnace system of claim 7 further comprising an auxiliary electrode heater disposed within the gas-tight CEVAR furnace chamber to heat the preheated silicon electrode during the CEVAR purification process.

11. The CEVAR furnace system of claim 7, the ingot withdrawal drive system further comprising a base upon which the bottom of the silicon ingot sits and a drive actuator connected to the base to control the rate of withdrawing of the silicon ingot from the short CEVAR open-bottomed crucible.

12. The CEVAR furnace system of claim 7, the base having a contour for interlocking contact with the bottom of the silicon ingot.

13. The CEVAR furnace system of claim 11 further comprising a DC power source having a first output and a second output connected between the preheated silicon electrode and the base or drive actuator.

14. The CEVAR furnace system of claim 7, the short CEVAR opened-bottom crucible having an

interior wall with a rectangular cross section, the interior height of the short CEVAR opened-bottom crucible being at least 60 percent and less than 120 percent of the length of the rectangular side of the silicon ingot formed in the CEVAR purification process.

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[0005] Generally the CEVAR process produces a purified metal by these four steps: (1) evaporating impurities as the metal electrode is melted and exposed to a vacuum in the CEVAR furnace; (2) floating out of the liquid (melted) metal impurities that have a lower density than the

[0019] The CEVAR purification process utilized in the present invention is generally similar to that described, for example, in the above disclosed prior art except for preheat of the silicon electrode used in the CEVAR process and the use of a short CEVAR open-bottomed, water-cooled metal crucible as disclosed herein. Generally for the present invention, during the CEVAR purification process, the preheated silicon electrode is placed in a short CEVAR open-bottomed crucible within the CEVAR furnace that is made gas-tight and brought to a vacuum or an otherwise controlled environment. During the process, direct current (DC current) flowing through the electrode and the melt formed below the electrode establishes an arc between the lower end of electrode and the top of the melt with the arc zone remaining within the height of the short CEVAR opened-bottom crucible so that a hot (at a temperature elevated above room temperature) solidified ingot exits the bottom of the short CEVAR opened-bottom crucible. Further controlled cooling of the hot solidified ingot exiting from the short CEVAR opened-bottom crucible is, as further described below, performed to substantially avoid cracking of the solidified ingot.

[0020] The ingot is withdrawn from the short CEVAR crucible into a heater system that provides controlled cooling over the temperature range in which the ingot is likely to crack. The ingot withdrawal rate is essentially matched to the ingot growth rate during steady state operation, so that the arc zone and the top of the ingot remain within the crucible. In an alternative arrangement of the present invention, the ingot is kept stationary and the crucible, electrode and the crucible's exit heater are raised together, to essentially match the growth rate of the ingot.

[0021] In practice of one embodiment of the present invention, the following process steps are performed: (1) forming an electrode from one or more pieces of silicon; (2) preheating the electrode to a temperature (by way of example and not limitation, between 800 and 1200 degrees Centigrade) at which it becomes sufficiently conductive (with a CEVAR process resistivity) to pass current in a subsequent CEVAR processing step without excessive voltage drop and prevention of cracking of the electrode ; (3) melting the electrode by a CEVAR process; (4) causing the resulting hot ingot to pass into a heating system adjacent to the open-bottomed CEVAR crucible while the ingot is at a sufficiently elevated temperature to prevent cracking; and (5) controlling the heating system so as to prevent cracking of the silicon ingot as it cools.

[0022] In alternative examples of the invention, the above preheating process step (2) may be performed within the CEVAR furnace or external to the furnace as described above.

CLAIMS

1. A method of producing a silicon ingot from a silicon electrode in a CEVAR purification process performed in a short CEVAR open-bottomed crucible disposed in a CEVAR furnace, the method comprising:

heating the silicon electrode to a heated temperature below the melting point of the silicon electrode prior to initiation of the CEVAR purification process to form a preheated silicon electrode having a CEVAR process resistivity;

melting the preheated silicon electrode by the CEVAR purification process for formation of a silicon ingot at an elevated temperature at the open bottom of the short CEVAR opened-bottomed crucible;

passing the silicon ingot at the elevated temperature through a heating system adjacent to the short CEVAR open-bottomed crucible; and

regulating the heating system to provide a temperature-controlled thermal environment for the silicon ingot at the elevated temperature as the silicon ingot exits the short CEVAR open-bottomed crucible to cool the silicon ingot without cracking.

2. The method of claim 1 where heating the silicon electrode is performed in an external heating chamber prior to placing the preheated silicon electrode in the CEVAR furnace and transfer of the preheated silicon electrode from the external heating chamber to the CEVAR furnace is accomplished in a controlled environment.

3. The method of claim 1 where heating the silicon electrode is performed subsequent to placing the silicon electrode within the CEVAR furnace.

4. The method of any one of claims 1 through 3 where heating the silicon electrode further comprises heating the silicon electrode to the heated temperature within the range of 800 to 1,200 degrees Centigrade to form the preheated silicon electrode.

5. The method of any one of claims 1 through 3 further comprising thermally insulating the preheated silicon electrode while melting the preheated silicon electrode by the CEVAR purification process.

6. The method of any one of claims 1 through 3 further comprising an auxiliary heater apparatus disposed within the CEVAR furnace to heat the preheated silicon electrode during the CEVAR purification process.

7. A CEVAR furnace system for producing a purified silicon ingot from a silicon

electrode, the CEVAR furnace system comprising:

a silicon electrode heating apparatus for preheating the silicon electrode to form a preheated silicon electrode;

a gas-tight CEVAR furnace chamber;

a short CEVAR open-bottomed crucible for containment of an arc zone from a CEVAR purification process melting the preheated silicon electrode, the short CEVAR open-bottomed crucible disposed in the gas-tight CEVAR furnace chamber;

a preheated silicon electrode drive system for lowering the preheated silicon electrode within the short CEVAR open-bottomed crucible as the lower end of the preheated silicon electrode melts in the CEVAR purification process;

an ingot heating apparatus disposed adjacent to the open bottom of the short CEVAR open-bottomed crucible through which the silicon ingot formed in the CEVAR purification process passes;

an ingot heating controller for controlling the ingot heating apparatus to provide a temperature-controlled thermal environment for the silicon ingot passing through the ingot heating apparatus; and

an ingot withdrawal drive system for alternatively withdrawing the silicon ingot from the short CEVAR open-bottomed crucible at a vertical growth rate of the silicon ingot during a steady state of the CEVAR purification process or raising the short CEVAR open-bottomed crucible, the preheated silicon electrode and the ingot heating apparatus at the vertical growth rate of the silicon ingot during the steady state of the CEVAR purification process.

8. The CEVAR furnace system of claim 7 wherein the interior height of the short CEVAR opened-bottom crucible is at least 60 percent and less than 120 percent of the diameter of the silicon ingot formed in the CEVAR purification process.

9. The CEVAR furnace system of claim 7 or 8 further comprising a vacuum lock chamber connected between the silicon electrode heating apparatus and the short CEVAR opened-bottom crucible in the gas-tight CEVAR furnace chamber to prevent exposure to air of the preheated silicon electrode during transfer from the silicon electrode heating apparatus to the gas-tight CEVAR furnace chamber.

10. The CEVAR furnace system of claim 7 or 8 further comprising an auxiliary electrode

heater disposed within the gas-tight CEVAR furnace chamber to heat the preheated silicon electrode during the CEVAR purification process.

11. The CEVAR furnace system of claim 7 or 8, the ingot withdrawal drive system further comprising a base upon which the bottom of the silicon ingot sits and a drive actuator connected to the base to control the rate of withdrawing of the silicon ingot from the short CEVAR open-bottomed crucible.

12. The CEVAR furnace system of claim 11, the base having a contour for interlocking contact with the bottom of the silicon ingot.

13. The CEVAR furnace system of claim 11 or 12 further comprising a DC power source having a first output and a second output connected between the preheated silicon electrode and the base or drive actuator.

14. The CEVAR furnace system of claim 7 or 8, the short CEVAR opened-bottom crucible having an interior wall with a rectangular cross section, the interior height of the short CEVAR opened-bottom crucible being at least 60 percent and less than 120 percent of the length of the rectangular side of the silicon ingot formed in the CEVAR purification process.