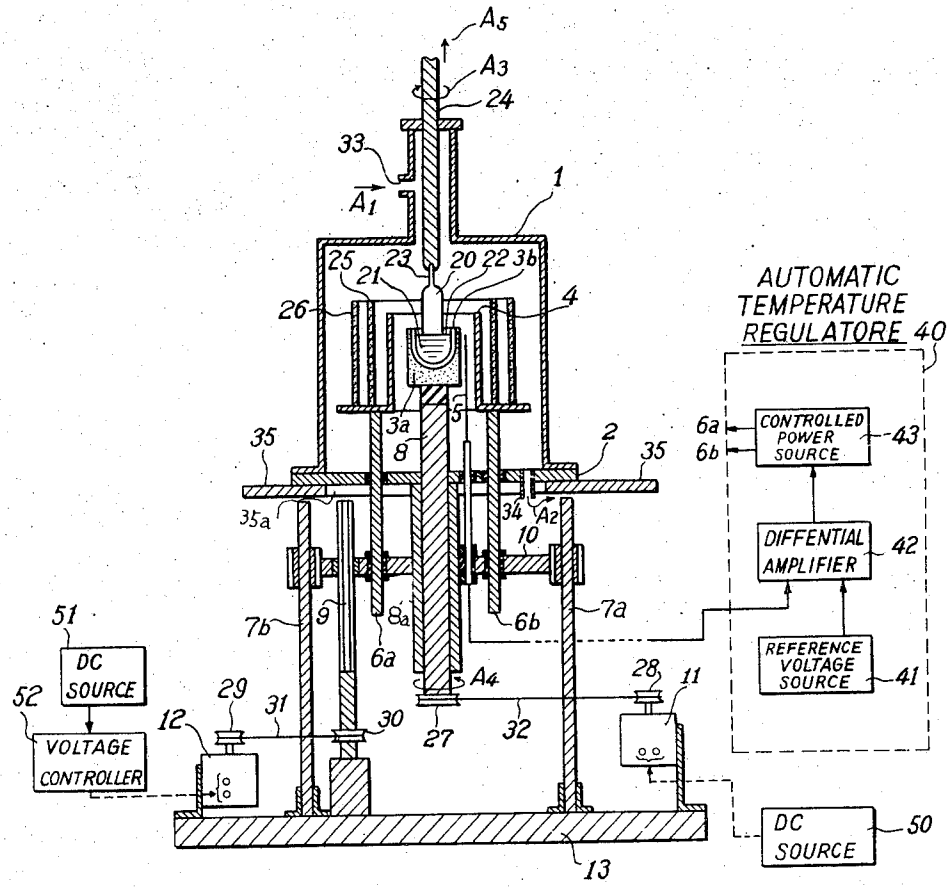
Oct. 10, 1972 SEIYA HIGASHI ET AL 3,700,412

CRYSTAL PULLING APPARATUS HAVING MEANS FOR MAINTAINING LIQUID-SOLID CRYSTAL INTERFACE AT A CONSTANT TEMPERATURE

Filed Nov. 26, 1969



United States Patent Office 3,700,412 Patented Oct. 24, 1972

3,700,412

CRYSTAL PULLING APPARATUS HAVING MEANS FOR MAINTAINING LIQUID SOLID CRYSTAL INTERFACE AT A CONSTANT TEMPERATURE

Seiya Higashi and Kenro Kobayashi, Tokyo-to, Japan, assignors to Kokusai Denki Kabushiki Kaisha, Tokyo-to, Japan

Filed Nov. 26, 1969, Ser. No. 880,111
Claims priority, application Japan, Dec. 6, 1968, 43/89,142
Int. Cl. B01j 17/18
U.S. Cl. 23—273 SP 9 Claims

ABSTRACT OF THE DISCLOSURE

Apparatus for growing crystals comprises a crucible for holding molten raw material to be grown into a crystal, means including a shaft for placing a seed crystal in the molten raw material to promote crystal growth, temperature sensing means including a movable thermocouple for measuring the temperature of the boundary interface existing between the grown crystal and the molten raw material and providing a corresponding temperature output signal, and temperature regulating means receptive of the temperature output signal for comparing same with a reference signal representative of the desired temperature which should be maintained at the boundary interface. The temperature regulating means comprises a differential amplifier for comparing the two signals and developing a difference signal which is applied to a controlled power source for controlling the energization of heating electrodes of a heater. In order to stabilize the temperature at the boundary interface, the thermocouple is mounted for movement integrally with the heater and in synchronization with the lowering movement of the boundary interface and actuating means is provided for effecting movement of both the thermocouple and heater in accordance with the lowering rate of the boundary interface to maintain the thermocouple at a constantly spaced-apart distance from the boundary interface thereby providing a more accurate temperature sensing function and resulting in a more stabilized crystal growth.

This invention relates to a crystal pulling device in which the developing crystal is gradually withdrawn from a melt of raw material, such as metal or semiconductor.

In case of producing crystals of metals or semiconductors of preferable characteristics by a crystal pulling method, it is very important to maintain stable the temperature of a melt of raw material and to maintain the temperature of a boundary plane between the developing crystal and the melt of raw material at a predetermined value peculiar to the raw material. By way of example, silicon which is a semiconductive material has a melting point of 1420 degrees in Celsius thermal scale, so that silicon assumes the solid state under the melting point and the liquid state above the melting point. Moreover, the diameter of the developing crystal is determined also by the thermal equilibrium at the boundary plane between the solid state and the liquid state. Accordingly, if the temperature of the raw material measured at the boundary plane cannot be maintained at a correct value, the diameter of the developing crystal cannot be maintained at a constant value. This will reduce the characteristic of the developed crystal. In the case of pulling a silicon crystal, it is necessary to maintain the temperature of the boundary plane within an extremely narrow temperature range, such as 1420±0.5 degrees in Celsius thermal scale.

To meet the above mentioned requirement by conventional arts, the temperature of the bottom or a part of a crucible containing the melt of raw material is measured

so that the measured temperature of the crucible is automatically controlled so as to be maintained at a constant value. In this method of conventional arts, however, since the quantity of the melt of raw material reduces in accordance with progress of the pulling, it is not avoidable that the temperature of the boundary plane deviates from the correct value even if the measured temperature of the crucible can be maintained at the constant value. If the measured temperature of the crucible is maintained at the contact value, the temperature of the boundary plane will assume an upward curve. Since the growth velocity of the developing crystal is determined by the temperature of the boundary plane between the developing crystal of solid state and the melt of the raw material, the diameter of the developing crystal of this case will be gradually reduced on account of a decrease of the growth velocity of the developing crystal. To maintain a uniform diameter of the developing crystal in conventional arts, a method of compensation had seen employed to lower down the measured temperature of the crucible. To perform this method of compensation, however, since it is necessary that an operator must adjust to successive preferable values the reference value of the automatic temperature control for the crucible in watching the diameter of the developing crystal, handling of this method of correction is troublesome and the quality of the produced crystal depends upon the pulling technique of the operator. Moreover, in a case where the quantity of the melt of raw material increases, the compensation technique of this method becomes more difficult since a time-lag from the adjustment of the measured temperature of the crucible to the correction of the diameter of the developing crystal is not negligible. Therefore, this method of compensation is not a reliable method to produce crystals having uniform preferable characteristics.

An object of this invention is to provide a crystal pulling device capable of developing a crystal having uniform preferable characteristics.

To attain the objects of this invention, a crystal pulling device of this invention, in which the developing crystal is gradually withdrawn from a melt of raw material contained ina crucible heated by automatically regulated heater means, has the following principles. In accordance with features of this invention, the sensing part of a temperature measuring means is provided at a position opposed to a boundary plane between the developing crystal and the melt of raw material to automatically regulate the temperature of the heater. The sensing part of the temperature measuring means is moved, at a constant very low speed or a controllable very low speed, in synchronism with the drop of the boundary plane caused by the growth of the developing crystal. Accordingly, the temperature of the boundary plane is stabilized and effectively maintained within a very narrow temperature range including a correct value peculiar to the raw material.

The principle, construction and operation of the crystal pulling device of this invention will be better understood from the following more detailed discussion in conjunction with the accompanying drawing, in which:

The single figure is a diagram including sectional view for illustrating an embodiment of this invention.

With reference to the drawing, an embodiment of this invention will now be described. This embodiment comprises a chamber **1** of stainless steel; a supporting plate **2** supported on a fixed plate **35** having a circular opening **35***a*; a crucible **3***a* of graphite; a crucible **3***b* of quartz supported on the crucible **3***a* and containing the melt **21** of raw material; heating means comprising an electrically energizable heating member in the form of a cylindrical heater **4**; temperature sensing means including a thermocouple **5** supported on a slider **10**; electrode **6***a* and **6***b* of the heater **4**; guide poles **7***a* and **7***b* for guiding the slider **10**; a pole

8 supporting the crucible 3*a* and rotatably supported in a tube 8*a* fixed to the supporting plate 2*a*; a geared pole 9 rotatably supported on a supporting plate 13 to drive the slider 10 upwardly and downwardly; the slider 10 supporting the electrodes 6*a* and 6*b* and the thermocouple or sensor element 5; a motor 11 for rotating the pole 8 through a grooved pulley 28, a rope 32 (e.g.; V-shaped leather rope) and a grooved pulley 27 fixed to the lower end of the pole 8; a motor 12 for rotating the geared pole 9 through a grooved pulley 29, a rope 31 (e.g.,; V-shaped leather rope) and a grooved pulley 30 fixed to the geared pole 9; and the supporting plate 13. At the outside of the heater 4, heat insulators 25 and 26 are provided. At the top of the chamber 1, a pulling pole 24 is rotatably supported. This pulling pole 24 is pulled upwardly as shown by an arrow A_5 while rotating in the direction of an arrow A_3 by means of suitable drive means (not shown) known per se. At the lower end of the pulling pole 24, a seed crystal 23 of raw material is fixed. Reference numerals 20 and 21 indicate respectively a developing crystal of solid state and a melt of raw material. Argon gas is injected from an opening 33 as shown by an arrow A_1 and exhausted from an opening 34 as shown by an arrow A_2. The output voltage of the thermocouple 5 is applied to one of two input terminals of a differential amplifier 42 and a reference voltage is applied to the other terminal from a reference voltage source 41. The output of the differential amplifier 42 is applied to a controlled power source 43. The power from the controlled power source 43 is applied to electrodes 6*a* and 6*b* to heat the heater 4. The reference voltage source 41, the differential amplifier 42 and the controlled power source 43 form an automatic temperature regulator 40 together with the thermocouple 5 and the heater 4 to maintain the boundary plane 22 between the crystal 20 and the melt 21 at a correct temperature. The automatic temperature regulator 40 operates so as to reduce the output of the differential amplifier 42 to the minimum. A DC source 50 is connected to terminals of the motor 11. A DC source 51 is connected to terminals of motor 12 through a voltage controller 52.

In this embodiment, the quartz crucible 3*b* supported on the graphite crucible 3*a* is rotated in accordance with the rotation of the rotating pole 8 rotated by the motor 11 as shown by an arrow A_4 but does not move upwardly or downwardly. Accordingly, the quartz crucible 3*b* is supported at a constant height with respect to the supporting plate 2. On the other hand, the heater 4 and the sensing part of the thermocouple 5 are moved downwardly in synchronism with the drop of the boundary plane 22 caused by the growth of the developing crystal 20 by actuating means. To perform this operation, the slider 10 supporting the thermocouple 5 and the heater 4 is moved downwardly by rotating the geared-pole 9 by the motor 12. The revolutions of the motor 12 is transferred, in reducing the revolutions, to the grooved pulley 30 through the grooved pulley 29 and the rope 31, so that the slider 10 is moved downwardly at a very low speed, such as 0.1 millimeter/minute. The time-speed diagram of this slider 10 can be determined by calculation or practical tests. In the usual case, the speed of lowering the slide 10 may be at a constant value, but this lowering speed may be deviated within a narrow range, such as one tenth of the regular lowering speed, by the voltage controller 52.

As mentioned above, the crystal pulling device of this invention has a feature in which the temperature detector (i.e.; the thermocouple 5) for the boundary plane 22 is moved downwardly in synchronism with the drop of the boundary plane 22 caused by the growth of the developing crystal 20. Since the temperature measured by the thermocouple 5 is maintained at a constant value by the automatic temperature regulator 40, the temperature of the boundary plane 22 can be always maintained at a very narrow temperature range including a correct value peculiar to the raw material.

In this case, the thermocouple 5 may be provided between the heater 4 and the thermal insulator 25 so that the sensing part thereof is opposed to the position of the boundary plane 22, since the temperature of a part of heater 4 opposed to the boundary plane 22 depends closely to the temperature of the boundary plane 22 as the results of our test.

Examples of our practical tests are as follows:

EXAMPLE 1

The inner diameter of the quartz crucible 3*b*	4 inches.
The raw material	Silicon.
The weight of the raw material	1.3 kilograms.
The diameter of the developing crystal	48 millimeters.
The pulling speed of the developing crystal	0.8 millimeters/minute.
The lowering speed of the slider	0.1 millimeters/minute.
The deviation of the diameter of the crystal	±4%.

EXAMPLE 2

The inner diameter of the quartz crucible 3*b*	6 inches.
The raw material	Silicon.
The weight of the raw material	4.3 kilograms.
Other condition and result	Similar to Ex. 1.

As mentioned above, since the temperature of the boundary plane between the developing crystal and the melt of the raw material can be stably maintained within a very narrow temperature range including a correct value peculiar to the raw material in the device of this invention, a crystal having uniform characteristics can be produced by the crystal pulling device of this invention.

What we claim is:

1. A crystal pulling device for gradually withdrawing a developing crystal from a melt of raw material, comprising: a crucible containing the melt of raw material; heater means for heating the crucible; temperature measuring means having a movable sensing part provided at a position closely opposed to a boundary plane existing between the developing crystal and the melt of raw material for measuring the temperature of the boundary plane and providing a corresponding output signal; means for automatically regulating the power supplied to the heater in accordance with a signal derived by comparing said output signal with a reference signal; and control means for effecting movement of both said sensing part of the temperature measuring means and said heater means together as an integral unit in synchronism with the lowering of the boundary plane caused by the growth of the developing crystal; whereby the temperature of the boundary plane is stabilized and maintained within a very narrow temperature range including a correct value peculiar to the raw material.

2. A crystal pulling device according to claim 1, wherein said temperature measuring means includes a thermocouple provided between the crucible and the heater means.

3. A crystal pulling device according to claim 1, wherein said temperature measuring means includes a thermocouple provided at the outside of the heater means heating the crucible.

4. A crystal pulling device according to claim 1, wherein said control means includes means for moving both said temperature measuring means and the heater means at a constant very low speed.

5. A crystal pulling device according to claim 1, wherein said control means includes means for moving both said temperature measuring means and the heater means at a controllable low speed.

6. In a crystal growing apparatus having a crucible for containing a molten raw material to be grown into a

crystal; and means for contacting the seed crystal with the molten raw material to effect growth of a crystal composed of the raw material upon the seed crystal; temperature sensing means including a movable sensor element disposed in spaced-apart opposed relationship to the boundary interface existing between the grown crystal and the molten raw material for sensing the temperature of said boundary interface and providing a corresponding temperature signal; temperature regulating means receptive of said temperature signal for comparing same with a reference signal representative of the desired temperature of said boundary interface and providing a resulting difference signal; heating means positioned in spaced-apart relationship from said crucible for applying heat energy to said crucible in accordance with said difference signal to thereby maintain the temperature of said boundary interface substantially at said desired temperature; and actuating means including means mounting said heating means and sensor element for movement together as an integral unit for effecting movement of both said sensor element and heating means in synchronization with the gradual movement of said boundary interface resulting from the progressive growth of the grown crystal accompanied by the progressive shrinkage of the volume of molten raw material contained in said crucible to maintain both said sensor element and heating means at a substantially constant distance from the boundary interface to accordingly stabilize the temperature of said boundary interface at said desired temperature.

7. A crystal growing apparatus according to claim 6; wherein said heating means comprises an electrically energizable heating member circumferentially disposed around said crucible; wherein said sensor element comprises a thermocouple; and wherein said actuating means includes means mounting said heating member and thermocouple for movement together as an integral unit.

8. A crystal growing apparatus according to claim 6; wherein said actuating means includes means for moving said sensor element at a variably set speed.

9. A crystal growing apparatus according to claim 6; wherein said actuating means includes means for moving both said heating means and said sensor element for movement together as an integral unit at a variably set speed.

References Cited

UNITED STATES PATENTS

2,890,139	6/1959	Shockley	23—273
2,979,386	4/1961	Shockley et al.	23—273
3,240,568	3/1966	Derby et al.	23—273
3,291,650	12/1966	Dohmen et al.	23—273
3,384,449	5/1968	Coin et al.	23—301
3,499,736	3/1970	Zwaneburg	23—273
2,948,799	8/1960	Weise	236—15 B
2,992,311	7/1961	Keller	23—301
3,192,302	6/1965	Keefer	236—15 B
3,241,925	3/1966	Cakenberghe	23—273
3,428,436	2/1969	Tucker	23—273
3,493,770	2/1970	Dessauer et al.	23—273

WILBUR L. BASCOMB, JR., Primary Examiner

R. T. FOSTER, Assistant Examiner

U.S. Cl. X.R.

236—15 B