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ZONE MELTING APPARATUS

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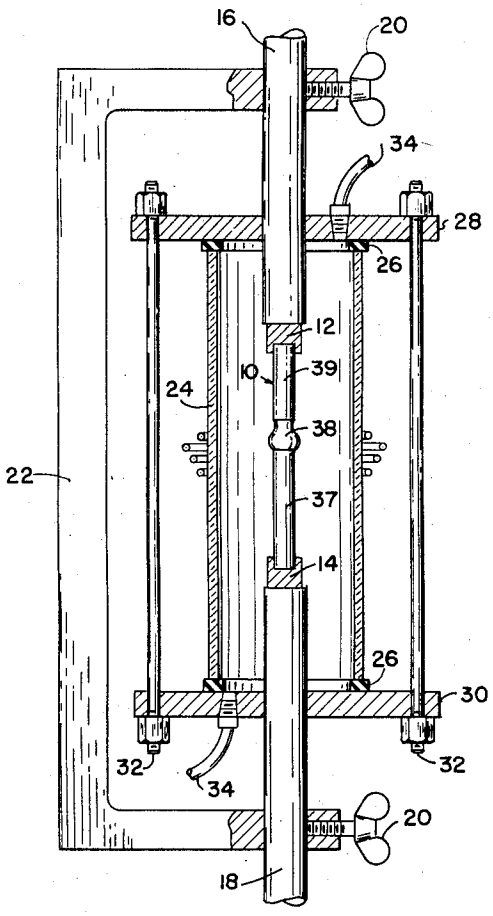


FIG. 1

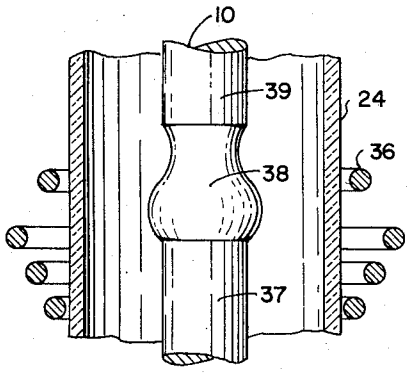


FIG. 2

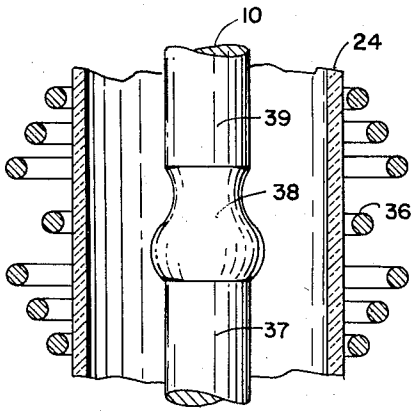


FIG. 3

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ZONE MELTING APPARATUS

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4 Claims. (Cl. 219—10.43)

My invention relates both to apparatus and methods for zone refining polycrystalline fusible semi-conductors in such manner as to avoid contamination thereof by foreign materials.

It is known that polycrystalline fusible semi-conductors, such as germanium or silicon, can be zone refined by vertically supporting an elongated body of said substance in a chamber, the body being spaced apart from the inner wall of the chamber; establishing within said body a molten zone which extends in cross section entirely through said body and which extends in length over a small fraction of the entire length of said body, the zone being held in a stable position by forces of surface tension; and causing said zone to traverse lengthwise at least a portion of said body by progressively melting the material of said body at one interface of said zone and progressively freezing the material of said zone at another interface of said zone.

This technique, known to the art as "floating zone crystallization," while sucessfully eliminating contamination by a foreign body, is at present limited in application to the formation of molten zones having small cross sectional areas. For example, if the molten zone cross sectional area of a silicon rod is increased beyond about 1 centimeter, the zone becomes unstable.

Stated differently, when the molten zone cross sectional area increases beyond a limiting value, the forces of surface tension cannot hold the zone in a stable position and the zone disintegrates.

I have invented apparatus and methods for using the floating zone technique in such manner as to increase the cross sectional area of a molten zone beyond that hitherto obtainable and yet maintain the stability of the zone.

Accordingly it is an object of the present invention to provide new and improved apparatus and methods of the character indicated.

Another object is to improve apparatus and methods for using the floating zone technique in such manner as to increase the cross sectional area of molten zones beyond that hitherto obtainable and yet maintain the zone in a stable condition.

Still another object is to partially counterbalance the gravitational force exerted on a molten zone, thus permitting the cross sectional area of a molten zone to be substantially increased without loss of stability.

Yet another object is to provide apparatus and methods for applying a levitational or buoyancy force to a molten zone.

These and other objects of my invention will either be explained or will become apparent hereinafter.

In accordance with the principles of my invention an elongated body of a fusible polycrystalline semiconductor, such as silicon or germanium, is end-supported in known manner. A first induction coil is mounted concentrically about the body, and when energized at a high frequency, establishes a molten zone within the body.

Under these conditions, the zone will adjust itself symmetrically about a plane passing through the center of the first coil and perpendicular to the first coil axis, a first zone interface being positioned above this plane, a second zone interface being positioned below this plane. Further, a second induction coil is mounted concentrically about the body adjacent but below the first coil. The second coil has a frustro-conical shape, the base of the second coil being adjacent the first coil. The second coil, when energized at a second and lower frequency is electromagnetically coupled to the molten zone but is not coupled to the solid portion of the body. The induction field established about the second coil exerts an upward or levitational magnetic force on the zone which renders same more buoyant and hence permits the cross sectional area of the zone to be substantially increase over that hitherto obtainable without loss of stability.

The stability of the above system can be enhanced by providing a third coil concentrically mounted about the body but above the first coil, the third coil also having a frustro-conical shape, the base of the third coil being adjacent the first coil. The second coil and the third coil are both energized at the same frequency.

The electromagnetic field established about the second and third coils, by virtue of their frustro-conical shape, not only exerts a levitational force, but also exerts lateral magnetic compressive forces upon the molten zone, thus providing an additional stabilizing action. This additional stabilizing action is necessary for zones of relatively large cross sectional areas. However, when it is advantageous to only increase the cross sectional area of a molten zone by a moderate amount, as compared to the area obtainable without use of levitational forces, the second coil, and when used the third coil, can be of the same shape as the first coil; i.e. it will have one or more helical turns.

Illustrative embodiments of my invention will now be described with reference to the accompanying drawings wherein:

Fig. 1 is a cross sectional view of apparatus in accordance with the invention and utilizing first and second induction coils;

Fig. 2 is an enlarged detail view showing the relative positions of the first and second induction coils of Fig. 1 with respect to the molten zone; and

Fig. 3 is an enlarged detail view showing the relative positions of first, second and third induction coils with respect to the molten zone.

Referring now to Fig. 1, an elongated body of a polycrystalline fusible semiconductor **10**, in this example silicon, is vertically held in place by sample holders **12** and **14** secured to its ends. Holder **12** is secured to an upper water cooled tube **16**, and holder **14** is secured to a lower water cooled tube **18**, tubes **16** and **18** being held in position by thumb screws **20**, these screws being mounted within a connecting bridge **22** insulatedly separated from tubes **16** and **18**. Tubes **16** and **18** are formed from copper or other equivalent metal.

The body **10** is mounted within a chamber formed by a quartz glass cylinder **24** sealed at its ends by gaskets **26**, top plate **28**, and bottom plate **30**. The chamber is held together by connecting rods **32**.

Inlet and outlet gas connections **34** in the plates **28** and **30** serve to feed an inert gas through the chamber or, alternatively, to evacuate the chamber.

A first induction coil **36** is positioned concentrically about the outside of cylinder **24**. This coil, when energized in conventional manner by an induction generator (not shown), at a frequency falling within the range 3–5 megacycles, heats a localized section of body **10** and produces a molten zone **38** in body **10**. The zone **38** has upper and lower liquid-solid interfaces **40** and **42**.

A second induction coil 44 having a frustro-conical shape is circumferentially disposed about body 10 and is positioned below coil 36 with its base being adjacent liquid-solid interface 42. This second coil 44 is energized by conventional manner at a frequency of about 500 kilocycles; the electromagnetic field produced about coil 44 is coupled to the molten silicon but due to its relatively low frequency is not coupled to solid silicon.

Consequently, the electromagnetic field about coil 44 exerts an upward or levitational force on the molten zone 38 (since the zone is free to move and the coils are held in fixed positions) which renders the zone more buoyant and permits the cross sectional area of a stable molten silicon zone, for example, to be increased from 1 centimeter to about 1 inch without the zone becoming unstable.

The molten zone is caused to traverse the body 10 lengthwise by loosening the thumb screws 20 and moving tubes 16 and 18 upward through the chamber.

Fig. 2 is an enlarged detail view showing the relationship between coils 36 and 44 and zone 38 for the apparatus of Fig. 1.

Additional zone stability with consequent increase in cross sectional area can be obtained, as shown in the enlarged detail view of Fig. 3 by circumferentially disposing a third coil 50 having a frustro-conical shape about body 10 above coil 36, the base of coil 50 being positioned adjacent solid-liquid interface 40 of zone 38. Coil 50 is energized in the same manner as coil 44.

In order to heat silicon effectively by induction, the frequency of the induction current must be increased over that normally used for metals. I have found that the frequency of about 3–5 megacycles is quite satisfactory, the power required being on the order of several kilowatts. Due to the high resistivity of silicon at room temperature, initiation of induction heating is sometimes difficult, and I have found it convenient to preheat the silicon (thereby reducing its resistivity) as, for example, by passing a current through tubes 16 and 18 and body 10.

While I have shown and pointed out my invention as applied above, it will be apparent to those skilled in the art that many modifications can be made within the scope and sphere of my invention as defined in the claims which follow.

What is claimed is:

1. An apparatus for zone refining an elongated body of a polycrystalline fusible semiconductor comprising means to end-support said body in a controlled atmosphere; a first induction coil circumferentially disposed about a portion of said body intermediate its ends, said coil, when energized at a first frequency, establishing within said body a molten zone which extends in length over a small fraction of the entire length of said body and which extends in cross section entirely through said body, said zone being disposed symmetrically about a plane perpendicular to the axis of said coil and passing through the center of said coil; a first liquid-solid interface of said zone being positioned above said plane, a second liquid-solid interface of said zone being positioned below said plane; a second induction coil having a frustro-conical shape and circumferentially disposed about said body, the base of said second coil being adjacent said second interface and downwardly displaced therefrom, the top of said second coil being below its base, said second coil, when energized at a second and lower frequency, being electromagnetically coupled to said zone and exerting a levitational force thereon, said zone being held stably in position by said levitational force and by surface tension.

2. An apparatus for zone refining an elongated body of a polycrystalline fusible semiconductor comprising means to end-support said body in a controlled atmosphere; a first induction coil circumferentially disposed about a portion of said body intermediate its ends, said coil, when energized at a first frequency, establishing within said body a molten zone which extends in length over a small fraction of the entire length of said body and which extends in cross section entirely through said body, said zone being disposed symmetrically about a plane perpendicular to the axis of said coil and passing through the center of said coil; a first liquid-solid interface of said zone being positioned above said plane, a second liquid-solid interface of said zone being positioned below said plane; and second and third induction coils having frustro-conical shapes and circumferentially disposed about said body, the bases of said second and third coils being respectively adjacent said second and first interfaces and respectively downwardly and upwardly displaced from the corresponding second and first interfaces, the tops of said second and third coils being respectively positioned below and above the corresponding bases, said second and third coils, when energized at a second and lower frequency, being electromagnetically coupled to said zone and exerting a levitational force thereon, said zone being held stably in position by said levitational force and by surface tension.

3. An apparatus for zone refining an elongated body of a polycrystalline fusible semiconductor comprising means to end-support said body in a controlled atmosphere; a first induction coil circumferentially disposed about a portion of said body inetrmediate its ends, said coil, when energized at a first frequency, establishing within said body a molten zone which extends in length over a small fraction of the entire length of said body and which extends in cross section entirely through said body, said zone being disposed symmetrically about a plane perpendicular to the axis of said coil and passing through the center of said coil; a first liquid-solid interface of said zone being positioned above said plane, a second liquid-solid interface of said zone being positioned below said plane; a second induction coil having a frustro-conical shape and circumferentially disposed about said body, the base of said second coil being adjacent said second interface and downwardly displaced therefrom, the top of said second coil being below its base, said second coil, when energized at a second and lower frequency, being electromagnetically coupled to said zone and exerting a levitational force thereon, said zone being held stably in position by said levitational force and by surface tension; and means to displace said zone in an axial direction along said body whereby said zone traverses at least a portion of said body.

4. An apparatus for zone refining an elongated body of a polycrystalline fusible semiconductor comprising means to end-support said body in a controlled atmosphere; a first induction coil circumferentially disposed about a portion of said body intermediate its ends, said coil, when energized at a first frequency, establishing within said body a molten zone which extends in length over a small fraction of the entire length of said body and which extends in cross section entirely through said body, said zone being disposed symmetrically about a plane perpendicular to the axis of said coil and passing through the center of said coil; a first liquid-solid interface of said zone being positioned above said plane, a second liquid-solid interface of said zone being positioned below said plane; and second and third induction coils having frustro-conical shapes and circumferentially disposed about said body, the bases of said second and third coils being respectively adjacent said second and first interfaces and respectively downwardly and upwardly displaced from the corresponding second and first interfaces, the tops of said second and third coils being respectively positioned below and above the corresponding bases, said second and third coils, when energized at a second and lower frequency, being electromagnetically coupled to said zone and exerting a levitational force thereon, said zone being held stably in position by said levitational force and by surface ten-

sion; and means to displace said zone in an axial direction along said body whereby said zone traverses at least a portion of said body.

References Cited in the file of this patent

UNITED STATES PATENTS

2,686,864 Wroughton et al. ---------- Aug. 17, 1954
2,686,865 Kelly ---------- Aug. 17, 1954
2,743,199 Hull et al. ---------- Apr. 24, 1956
2,773,923 Smith ---------- Dec. 11, 1956
2,792,317 Davis ---------- May 14, 1957