Dec. 12, 1972 W. KELLER 3,705,789

METHOD FOR FLOATING ZONE MELTING

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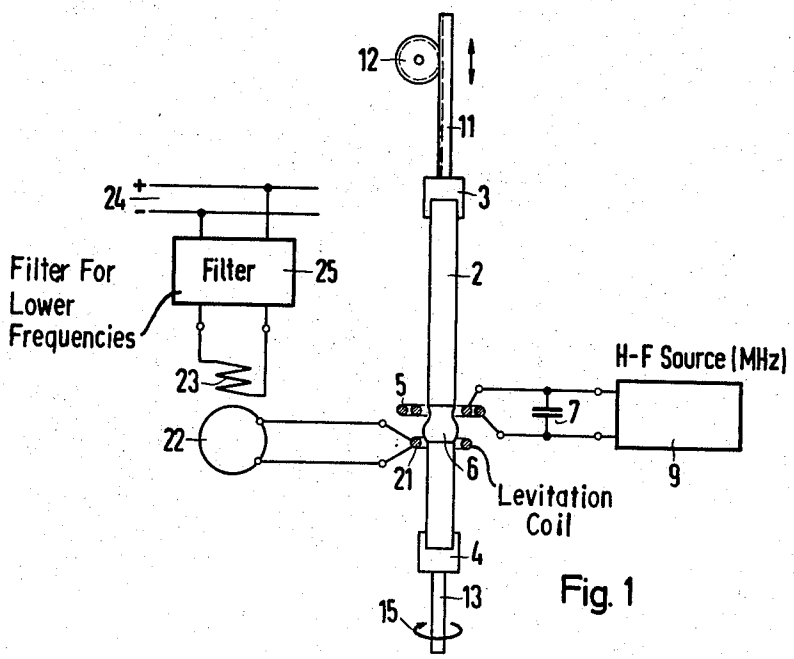


Fig. 1

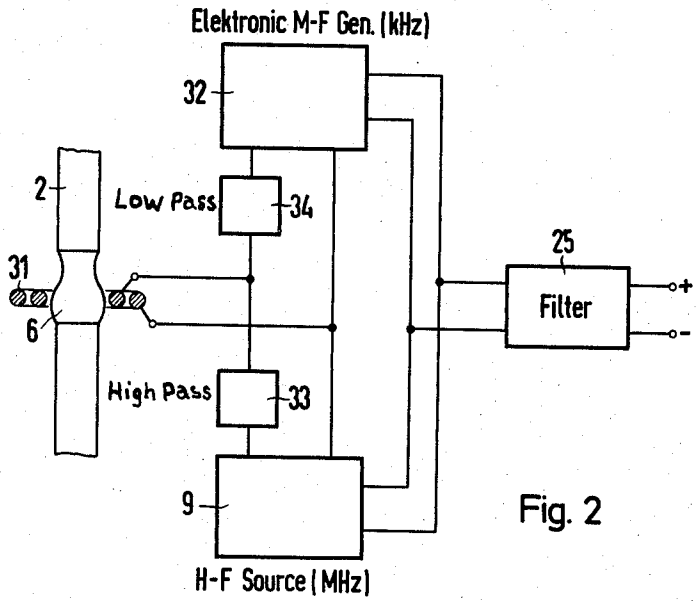


Fig. 2

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METHOD FOR FLOATING ZONE MELTING

Wolfgang Keller, Pretzfeld, Germany, assignor to Siemens Aktiengesellschaft, Berlin and Munich, Germany

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ABSTRACT OF THE DISCLOSURE

Floating zone melting of a vertically mounted rod is performed while energizing a levitating field winding by alternating current of medium frequency (500 Hz.–500 kHz.) and maintaining the low-frequency (below 300 Hz.) amplitude fluctuations of the energizing current below 5%, preferably below 1%. Used as current source is an alternating-current generator whose excitation or feeder direct current is smoothed by electronic filtering.

My invention relates to the float-zone melting of crystalline rods, preferably rods of semiconductor material.

For this purpose, the rod is held at both ends in a vertical position. The two rod holders are fastened to coaxial vertical shafts of which one or both can be kept in continuous rotation about the shaft axis. The holder shafts may be shifted in the vertical direction relative to one another if this is needed for controlling the cross section of the rod during the zone-melting operation, especially for such purposes as precisely maintaining a constant cross section or changing the cross section in a predetermined manner. The molten zone of the rod, having a very short axial length in comparison with the total axial length of the rod being processed, is produced by means of a heating device, for example an induction heater winding which coaxially surrounds the rod and is energized by high-frequency current. By relative longitudinal displacement between heating device and rod, the molten zone is caused to travel lengthwise through the rod. In most cases such crucible-free or floating zone melting serves to purify the rod material and to also convert it to a monocrystal with the aid of a crystal seed fused to one rod end. An apparatus suitable for the operation just described is illustrated, for example, in U.S. Pat. 3,189,415.

It has been found that when crucible-free zone melting is applied to rods of large diameters, such as about 30 to 40 mm. or more, the molten zone often is instable and tends to drain away or to solidify. Furthermore, a uniform growth of the material crystallizing out of the molten zone is not readily secured. In some cases the rod, after zone melting, exhibits noticeable bulges. To minimize such impairments, a levitating magnetic field has been applied to the sagging bottom portion of the molten zone. Such a field can be brought about as follows.

The induction heater winding often employed as heating device for floating zone melting is energized by high-frequency current of a few megahertz. The field of this coil also imparts some supporting or levitating effect upon the molten zone. However, it is known to additionally support the molten zone by producing a magnetic field with the aid of an induction coil energized by alternating current of much lower frequency, namely between 500 Hz. and 500 kHz. The induction heater winding itself can also be used for producing an additional stabilizing alternating field by energizing this winding not only with high-frequency current but also with a low to medium-frequency current. While by any of these means the desired levitating effect can be obtained, thus preventing the molten zone from excessively bulging or draining away, the occurrence

of an appreciable and usually not controllable number of dislocations in the processed rod has been inevitable.

It is an object of my invention to further improve zone-melting methods and apparatus of the above-mentioned kind toward obtaining products of better crystalline qualities.

More specifically it is an object of the invention to provide during zone-melting operations for the desired levitating effect and securing a product that exhibits considerably fewer imperfections, especially dislocations, than heretofore encountered.

To achieve these objects, and in accordance with the discovery which constitutes my invention, I perform a float zone-melting operation basically in the above-described manner, namely by melting an axially narrow zone of a vertically mounted rod and simultaneously moving the zone axially through the rod while supporting the molten zone by the magnetic field of a coaxial induction coil energized by alternating current in the above-mentioned frequency range of about 500 Hz. to about 500 kHz. According to the essential feature of my invention, however, I maintain the low-frequency amplitude fluctuations of the energizing alternating current, these fluctuations being in the range up to about 300 Hz., especially of about 100 to 300 Hz. below a maximum limit of 5% of the normal amplitude. Preferably the low-frequency amplitude fluctuations of the alternating current supplied to the levitating coil are maintained below the limit of 1% during the entire zone-melting operation.

I have discovered that by proceeding in this manner the number of dislocations in the finished crystal is greatly reduced. This can be explained by assuming that the low-frequency amplitude fluctuations contained in the output voltages of the conventionally used generators, cause a corresponding vibration or jarring of the melt, thus disturbing the crystalline growth.

The reduction in amplitude fluctuations according to the invention is secured, for example, as follows. When supplying the levitating coil, which may either be the induction heater itself or a separate levitating coil, with alternating current from an electronic generator operating with electronic tubes or semiconductor devices, the direct-current feed voltage employed for operating the alternating-current generator is supplied thereto through a filter circuit so as to become smoothed to the extent needed for minimizing the alternating-current amplitude fluctuation down to less than 5%, preferably less than 1%. When supplying the alternating current to the levitating coil from a dynamo-electric machine, the direct-current feed voltage employed for providing field excitation in the alternator may be passed in the same manner through a smoothing filter.

The invention will be further described with reference to the accompanying drawing in which:

FIG. 1 shows schematically an apparatus and appertaining circuitry for performing the method of the invention; and

FIG. 2 is a schematic diagram of modified apparatus for the purpose of the invention.

In FIG. 1 there is shown a rod-shaped body 2 of semiconductor material such as silicon, germanium or the like. The rod is vertically mounted between two holders 3 and 4. A heating coil 5 surrounds the rod and produces a melting zone 6 by induction heating. The heating coil 5 can be moved axially along the rod 2 at a given speed and is connected in parallel relation to a capacitor 7 to form an inductive heating or tank circuit therewith. This circuit is connected to the output terminals of a high-frequency generator 9 furnishing a current of a few mHz. The holder 3 carries a coaxial rack 11 meshing with a pinion 12 by means of which the holder 3 can be moved vertically

toward or away from the holder **4** which is not displaceable in the vertical direction. The holder **4** is joined with a coaxial shaft **13** which permits rotating the holder **4** and the rod portion attached thereto, the rotation being schematically indicated by an arrow **15**.

The rod **2** is further surrounded by a supporting or levitating coil **21** which is close to the lower solid-to-liquid boundary of the molten zone **6**. The coil **21** is mechanically joined with the heater winding **5** to move together therewith. The means for displacing the coils are not illustrated. In this respect, reference may be had, for example, to the above-mentioned Pat. No. 3,189,415. The levitating coil **21** is shown connected to a dynamo-electric alternator **22** whose excitation winding **23** receives direct current from buses **24** through a smoothing filter so that, for example, the 100 Hz. fluctuations of the alternator voltage amount to less than 1% of the amplitude.

In the embodiment illustrated in FIG. 2, the zone-melting apparatus may correspond to the one described above with reference to FIG. 1, except that only a single winding **31** is provided to serve simultaneously as induction heater and supporting coil. The winding **31** is connected by means of a high frequency pass **33** to a high-frequency source **9** which provides the heating current proper, and is also connected by means of a low frequency pass **34** to an electronic medium-frequency generator **32** which furnishes a frequency, for example of 10 kHz., to provide the desired levitating action. The direct-current feed voltage for operating the electronic generator **32** is supplied from a direct-current source through a filter **25** as described above. The same filtered direct current may also be applied to the source **9**, if desired.

It has been found that by thus supplying the feed or excitation current to the alternating-current generator which energizes the levitating field of the zone-melting equipment, the above-mentioned disturbances in the growth of the crystal resulting from the molten zone can be greatly suppressed, in many cases down to invisibility.

In one example of such operation, the excitation current of a 10 kHz. machine alternator having an output power of 10 kw. was filtered by means of conventional electronic filter circuits so that the 100 Hz. fluctuations of the alternating output voltage amounted to less than 1%. This was found to be particularly advantageous for the zone

melting of relatively thick rods narrowly surrounded by the coil for producing the supporting or levitating field. The rods thus zone-melted exhibited a smooth surface free of transverse grooves or striations otherwise observed.

To those skilled in the art it will be obvious upon a study of this disclosure that with respect to mechanical equipment and circuitry my invention permits of various modifications and hence may be given embodiments other than particularly illustrated and described herein, without departing from the essential features of my invention and within the scope of the claim annexed hereto.

I claim:

1. In the method of crucible-free zone melting semiconductor and other crystalline rods by melting an axially narrow zone of a vertically mounted rod and simultaneously moving the zone axially through the rod while supporting the molten zone by the magnetic field of a coaxial induction coil energized by alternating current, the improvement which comprises supplying to the coil an energizing alternating current of about 500 Hz. to about 500 kHz. frequency and maintaining low-frequency (below 300 Hz.) amplitude fluctuations of said alternating current below a maximum limit of 5% and which further includes electronically generating said coil current with the aid of direct feed current, and filtering said feed current to thereby maintain said fluctuations below said limit.

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NORMAN YUDKOFF, Primary Examiner

R. T. FOSTER, Assistant Examiner

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