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METHOD FOR INTERIOR ZONE MELTING OF A CRYSTALLINE ROD

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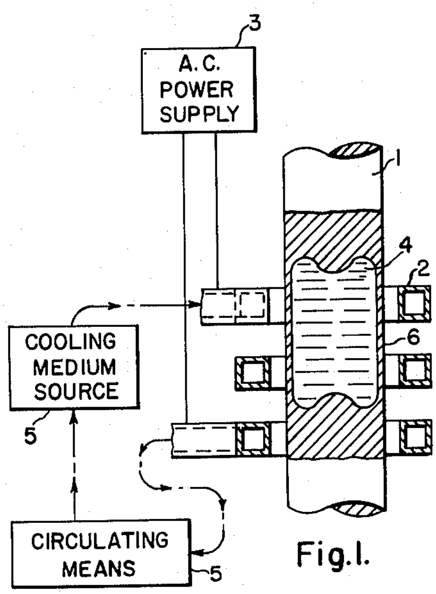

Fig.1.

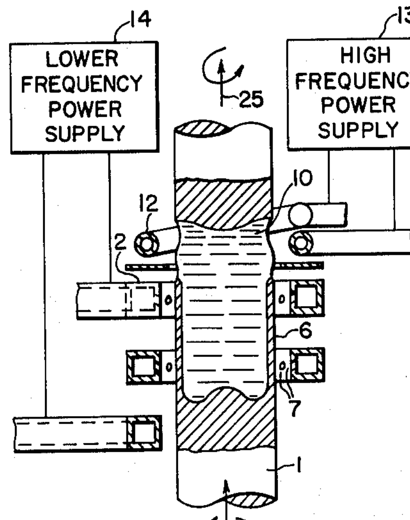

Fig.2.

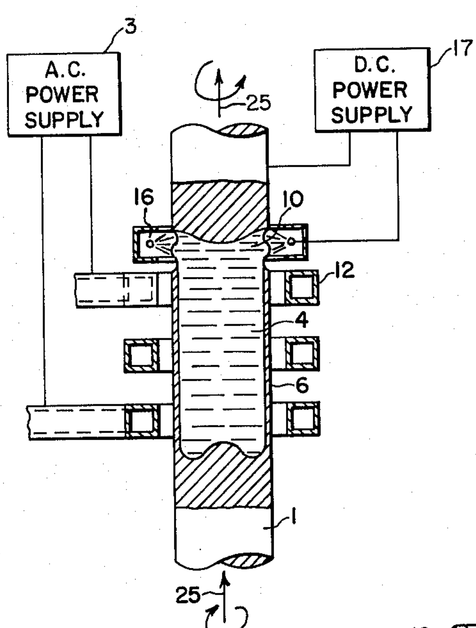

Fig.3.

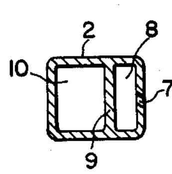

Fig.5.

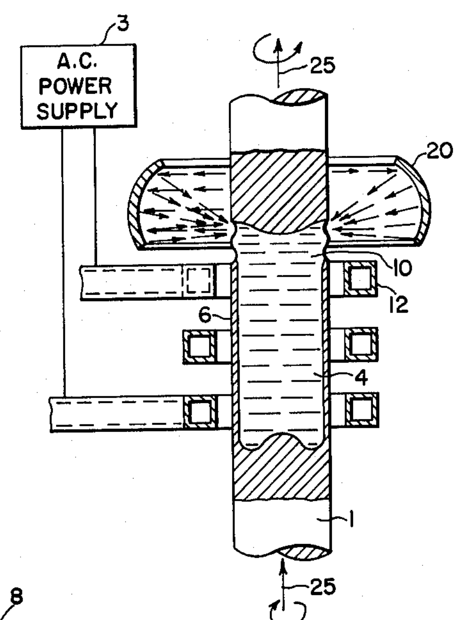

Fig. 4.

WITNESSES

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METHOD FOR INTERIOR ZONE MELTING OF A CRYSTALLINE ROD

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The present invention relates to a method and apparatus for zone melting, and more particularly to a method and apparatus for crucible-free floating-zone melting of elongated material such as semiconductor material, refractory metal, etc. in rod-shaped form, according to which a longitudinal molten zone is caused to move axially along the material while it is held in a vertical attitude.

Various techniques are employed to obtain the molten state of the longitudinal zone of the rod material, such, for example, as by electron bombardment heating and induction heating. The liquefied material of a through-melted or floating molten zone between the adjacent upper and lower solid portions of the rod is believed to be retained between such portions primarily by surface tension, although in the case of heating by induction a levitating influence from the magnetic field of the coil can be obtained from a specially-designed induction coil to contribute to confinement of such molten zone. In any event, the practice has been to subject a selected lengthwise zone of the rod material to a single source of heat, such as a cathode coil for electron bombardment heating or an induction coil for induction heating, for a sufficient period of time to through-melt the rod. In accord with the usual practice, design and operating parameters are chosen to produce the molten zone in such previous fashion while causing such zone to scan the rod vertically upward by effecting relative axialwise movement between the rod and an encircling induction heating coil or an encircling electron bombardment heating coil This practice is entirely satisfactory in many respects, but has resulted in limitation of the maximum diameter of rod which may be floating zone-melted without spill-over from the uniformly through-melted zone. For silicon rod, for example, this maximum diameter with induction heating and levitating techniques appears to be 1¼ inches, and for tungsten rod, for another example, it appears to be considerably less.

In view of the foregoing remarks, it becomes a prime object of the present invention to provide improvements in the aforedescribed method and apparatus whereby larger diameter rod-shaped material may be floating zone melted.

In accord with such object, general features of a longitudinal zone of the invention includes the melting of the rod material internally by one heat source, while an outer solidified cup-shaped or bowl-shaped shell is maintained in the rod material in containment of a considerable portion of the inner molten material, and the melting of the rod externally by another heat source to give a complete through-melting only at the upper edge of the shell and at the top of the molten zone for a sufficiently narrow through-melted axial distance to maintain the molten material confined radialwise between the bottom of the upper solidified portion of the rod and the upper unmelted rim of the solidified shell. Such internal and external melting of the rod should be effected simultaneously By virtue of this novel technique, the axial length of the floating or through-melted part of the molten zone may be kept sufficiently narrow to assure containment of a larger diameter zone than heretofore possible and therefore a large diameter rod can be floating zone melted than heretofore. It will be understood that the rod material is scanned by the molten zone as thus established, this scanning which per se forms no part of this invention, can be effected by any suitable means which obtains relative axialwise movement between the rod material and the heat sources. Rotation of the rod also is desirable and can be obtained in well known manner.

Other features, objects, and advantages of the invention will become apparent from the following detailed description taken in connection with the accompanying drawings in which:

FIGURE 1 illustrates in elevation, partly in outline and partly in section, the internal heating effect of a liquid-cooled induction heating coil on a rod-shaped material when functioning in accord with a feature of the invention;

FIGURE 2 illustrates the effect of melting the rod material internally with the liquid-cooled coil of FIGURE 1 at one power supply frequency, and melting through the outer periphery of the rod material by a second induction coil at a higher power supply frequency;

FIGURE 3 illustrates an alternate technique substituting electron bombardment heating in lieu of the external-heat induction coil of FIGURE 2; and

FIGURE 4 illustrates another alternate arrangement for obtaining the external heating which employs a reflected radiation technique; and

FIGURE 5 is a cross-sectional view of a turn of a combined liquid-cooled coil and cooling gas coil.

Referring now to the exemplification in FIGURE 1, in accord with a feature of the invention as discussed generally hereinbefore, the internal heating of the vertically-extending rod material is accomplished by an encircling multi-turn water-cooled, hollow-tubing, induction coil **2** which is energized from an alternating current power supply **3** by alternating current at a frequency and power level sufficient to heat a longitudinal section or segment of the rod and cause an inner region **4** of the zone to melt while an outer encircling solid wall **6** remains unmelted by such coil by virtue of dissipation of heat therefrom, as by radiation to the cooled coil **2** and its environs. A frequency of ten kilocycles per second, for example, at a power level of 40,000 watts with two-turn ¼" x ¼" square section hollow copper coil cooled to 80° F. will internally melt a .675 diameter tungsten rod with a .040 inch approximately thick outer wall **6**. Considerably larger diameter rods of tungsten or other refractory metal, similarly can be internally melted with suitable choice of frequency, power level, and coil temperature.

The forming of the outer wall **6** in encirclement of the inner region **4** of molten zone of the rod **1** is dependent upon dissipation of heat from the surface of the rod, as by radiation to its environment and this rate is a function of the absolute temperatures to the fourth power between rod **1** and such environment. It will be seen that the higher the melting point of the rod material, the greater will be the capability for heat to radiate from its outer surface and facilitate realization of the desired surface cooling effect by radiation alone. For example, tungsten which has a melting point of about 6170° F. (3410° C.) readily radiates sufficient heat to a coil and housing even at 100° F. to obtain the solid-shell molten-interior encasement effect, which would be true also of other refractory metals such as molybdenum. Material such as silicon, which has a melting point of 2605° F. to appreciate the same result by radiation cooling effect alone may not be attainable even with cryogenic cooling of the coil **2**. In this case, surface cooling of the silicon rod within such coil may be aided by directing a flow of gas over the outer surface of the rod material as by way of the interior of the coil **2** and orifices **7** at the inner periphery of the coil turns, as in FIGURE 2, where such cooling gas may also serve to cool the coil, or via a separate gas manifold **8** formed separately or integrally with the coil **2** which may be separated by a partition **9** from an interior portion **10** of the coil through which cooling water is circulated in a well-known manner. Exemplified structural details of such a coil turn are shown in cross-section in FIGURE 5.

To effect the through-melting of the rod wall **6** at the top of the inner region **4** of the molten zone within a narrow axialwise portion **10** in accord with the invention as aforediscussed herein, different techniques may be employed. For example, as in FIGURE 2 by employment of a single-turn water-cooled induction coil mounted above the coil **2** and energized from a power supply **13** of higher frequency than that supply **14** from which the coil **2** is energized. The lower frequency energization of coil **2** performs the deeply penetrating heating effect for the interior melting, and the higher frequency energization of coil **12** concentrates its heating effect in the outer portion of the rod to melt through the wall **6**, such latter concentration of outer surface heating acting to overcome the heat dissipation effect of radiation from the rod. As exemplified in FIGURE 3, in lieu of coil **12**, it can be accomplished by electron bombardment heating by electron emission from an inwardly-directed filament turn **16** connected to one side of a high voltage D.C. power supply **17** which is connected at its opposite side to the rod **1**. Or, as exemplified in FIGURE 4, such surface-concentrated heating can result from re-radiation of heat from the rod back to the selected surface region by means of a reflector ring **20** of suitable focusing configuration. In all cases, it will be understood that relative movement between the rod **1** to be floating-zone melted, as for conversion from polycrystalline to monocrystalline form, and the coil **2** and auxiliary final-melt heat source **12**, **16** or **20** is required to effect progressive movement of the molten zone along the length of the rod, as is well-known. The rod **1** also is rotated during such progressive zone melting to obtain a uniform heating effect from the encircling heat creating sources and to visually confirm the creation of a thru-melt. Such movements may be effected in a manner as described and shown in our copending U.S. patent application, Serial No. 219,957, filed August 28, 1962, titled "Apparatus for Zone Heating," or in other suitable ways. It is assumed herein that the workpiece rod **1** is moved axially relative to the heating coil, as indicated by arrows **25** in the drawings. However, insofar as the present invention is concerned, relative axialwise movement may be obtained by movement of the heating coil as has been taught heretofore by others.

Although not shown in the drawings, the zone melting method of the present invention will be carrried out in an enclosure which may be an evacuable one, a vacuum being necessary in the technique exemplified structurally in FIGURE 3 employing electron bombardment, and which enclosure alternatively may contain various gases such as argon, helium, hydrogen, etc., in which case the gas for cooling the rod **1** as in FIGURE 2 would be recoverable for recirculation following suitable cooling and treatment for removal of contaminants.

While there have been shown and described what are at present considered to be the preferred embodiments, uses, and advantages of the invention, modifications thereto and other uses and advantages may readily occur to those skilled in the art. It is not desired, therefore, that the invention necessarily be limited to the specific arrangements shown and described, and it is intended to cover in the appended claims all such modifications and uses as fall within the true spirit and scope of such invention.

We claim as our invention:

1. In a method of floating-zone melting a rod-shaped material susceptible to floating-zone melting, in which the rod is vertically disposed and a molten zone is created in the rod and caused to move lengthwise therealong, the improvement for creating such molten zone comprising the steps of heating a longitudinal segment of the rod to obtain interior melting thereof while heat is dissipated from its outer surface to preserve an elongated solid outer shell in encirclement of the molten interior portion of the zone, and heating an axialwise narrow region of said shell at the upper portion of the molten zone to give through-melting of the rod exclusively at such narrow region.

2. In a method of floating-zone melting a rod of refractory metal material susceptible to floating-zone melting, in which the rod is vertically disposed and a molten zone is created in the rod and caused to move lengthwise therealong, the steps for creating such molten zone, inductively heating a longitudinal segment of the rod at a frequency and power level which causes melting of the interior thereof while dissipation of heat by radiation from the exterior preserves a solid outer wall which encircles the molten interior, such molten interior being of greater overall length than can be retained between adjacent upper and lower solid rod portions as a floating zone through-melted for substantially the entirety of such length, and additionally heating an axialwise region of said outer wall at the upper portion of the molten zone to give through-melting of the rod exclusively in a sufficiently narrow region to permit such retention of the molten material.

3. In a method of floating-zone melting of a vertically-extending rod-shaped material susceptible to floating-zone melting, the steps of creating the molten zone by melting a longitudinal segment of the interior of the rod while its outer surface remains solid except for a narrow through-melted region at the top of lesser axial length than the axial length of the overall molten zone, such molten interior being of greater overall length than can be retained between adjacent upper and lower solid rod portions as a floating zone through-melted for substantially the entirety of such lentgh, and causing the thus-shaped molten zone to be moved axially along the rod.

4. In a method of floating-zone melting of a rod-shaped material susceptible to floating-zone melting, in which the rod is vertically disposed and a molten zone is created in the rod and caused to move lengthwise therealong, the creating of such molten zone by the heating of a longitudinal segment of the rod while selectively cooling areas of its outer surface in said segment such that within the segment its interior becomes molten and its exterior remains solid except for a narrow through-melted region at the top of the molten-zone, such molten interior being of greater overall length than can be retained between adjacent upper and lower solid rod portions as a floating zone through-melted for substantially the entirety of such length.

5. In a method of floating-zone melting a floating-zone-meltable material in rod shape, in which the rod is vertically disposed and a molten zone is created in the rod and caused to move lengthwise therealong, the steps for forming the molten zone by penetratedly heating a longitudinal segment of the rod while providing for dissipation of heat from its outer surface, thereby creating an elongated interior of molten material encased in solid material, and surface heating of a narrow portion at the top of such molten interior to melt such encasing solid material and obtain through-melting of the molten zone exclusively at such narrow portion, such molten interior being of greater overall length than can be retained between adjacent upper and lower solid rod portions as a floating zone through-melted for substantially the entirety of such length.

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