

Sept. 15, 1959

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2,904,411

SUSPENSION OF LIQUID MATERIAL

Filed June 17, 1955

4 Sheets-Sheet 1

FIG. 1

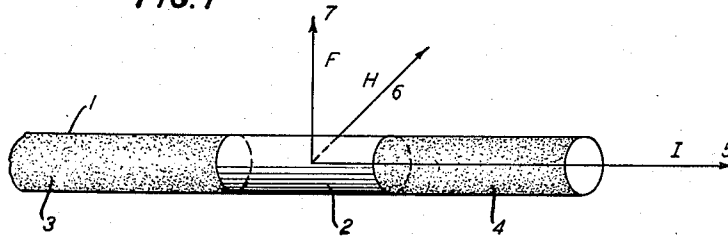
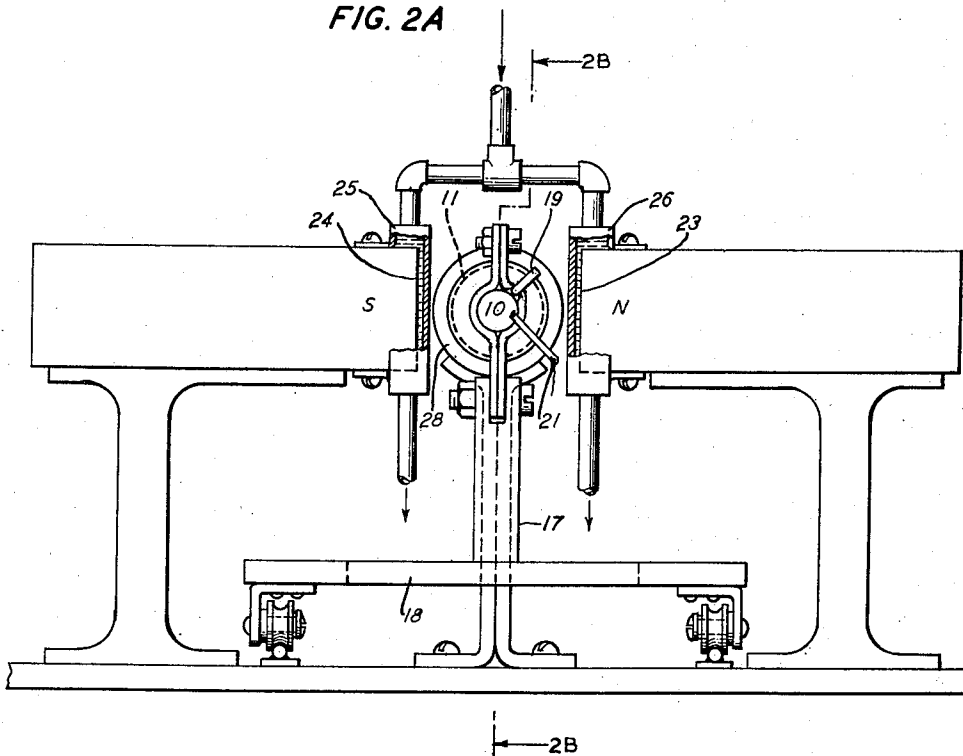


FIG. 2A



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FIG. 2B

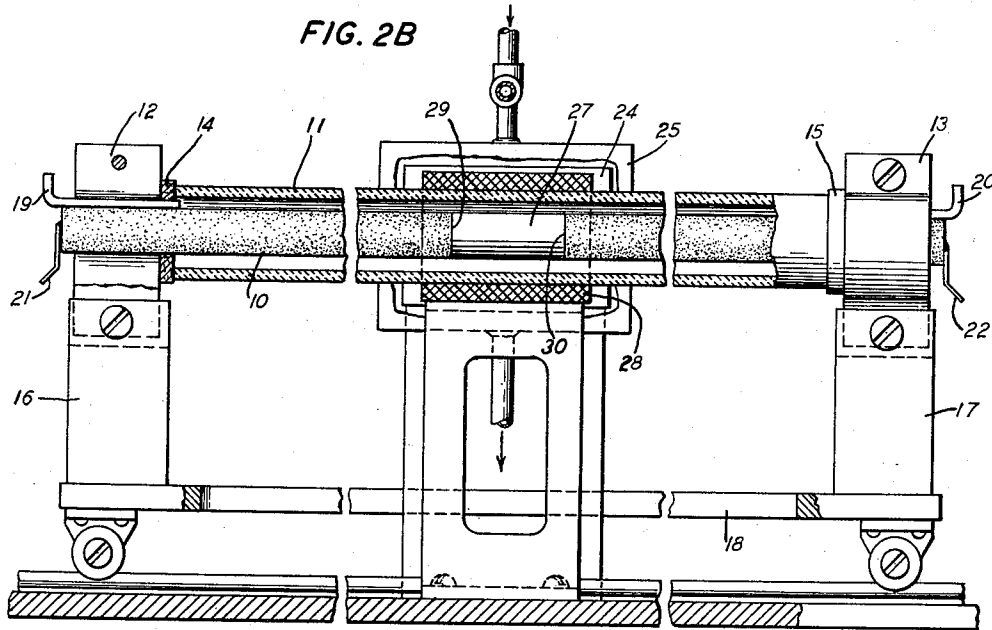


FIG. 3A

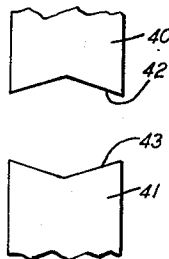


FIG. 3B

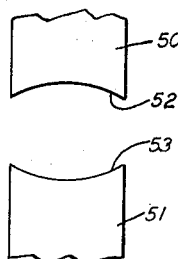


FIG. 3C

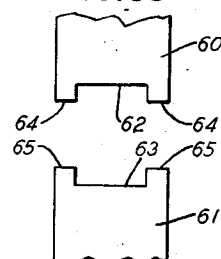


FIG. 4A

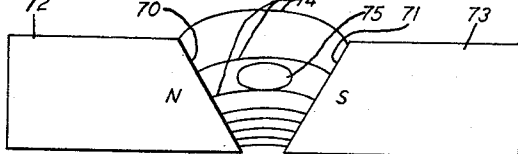


FIG. 4B

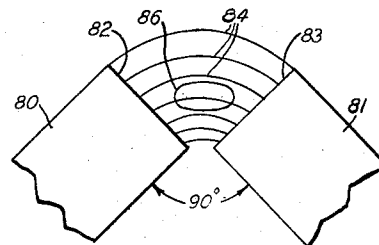
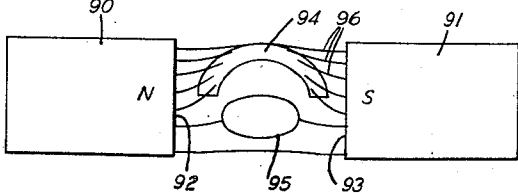


FIG. 4C



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FIG. 5A

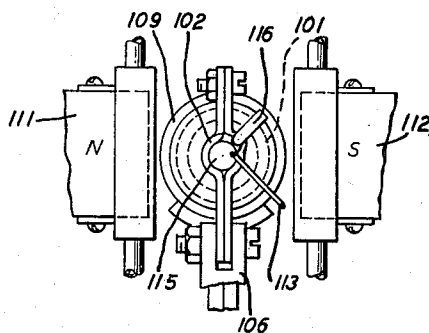
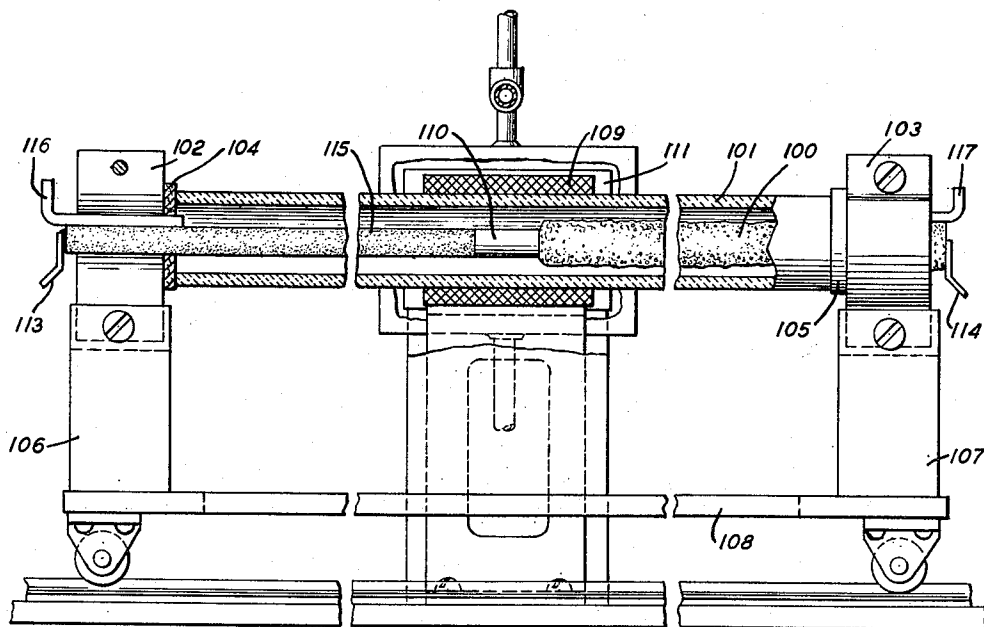


FIG. 5B



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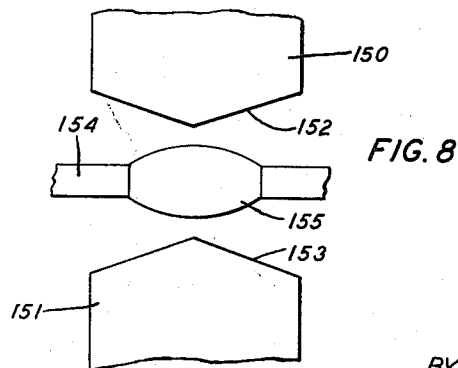
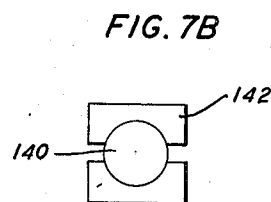
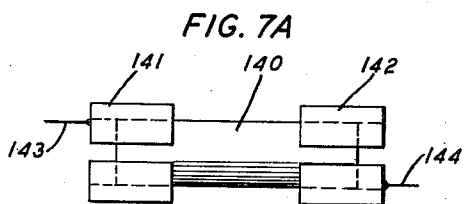
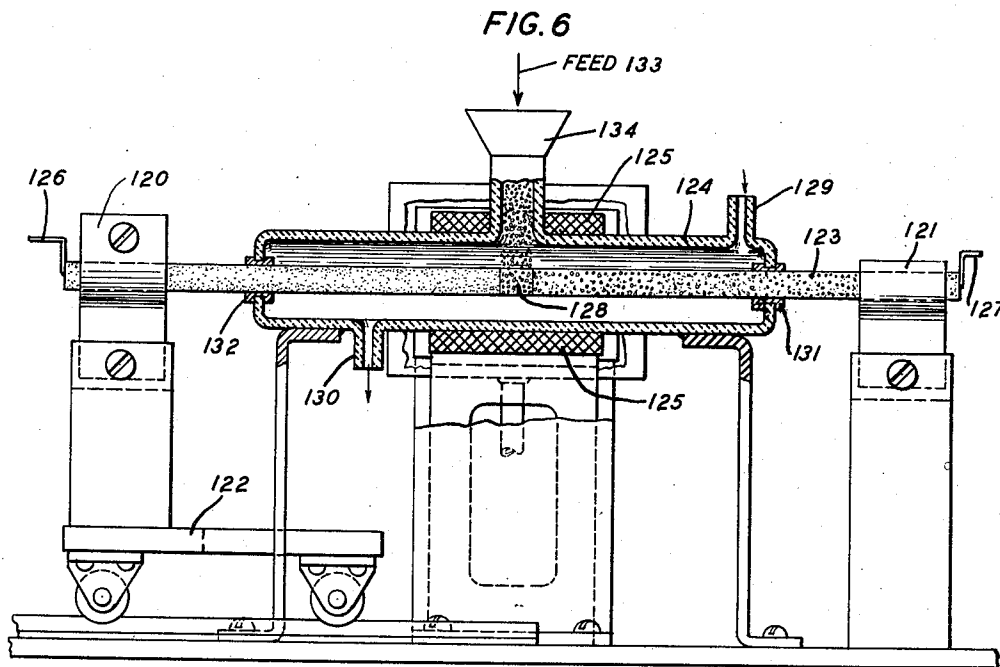
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4 Sheets-Sheet 4



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SUSPENSION OF LIQUID MATERIAL

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4 Claims. (Cl. 23—295)

This invention relates to methods of material treatment whereby liquid portions of such material are electromagnetically suspended during treatment. The processes of this invention are particularly applicable to the zone-melting processes as described in "Transactions of the American Institute of Mining and Metallurgical Engineers," volume 194, pages 747 through 753, 1952, and are valuable in the treatment of semiconductive materials such as germanium and silicon; reactive metals such as titanium, zirconium, iron, tantalum and beryllium, fused salts; and electrolytes having appreciable conductivity such as sodium chloride or potassium hydroxide aqueous solutions.

The processes of this invention are based on the reactive force which results when a magnetic field component is maintained across a body of material under treatment at an angle of 90 degrees to a current flow component through the body.

In the processes of this invention, a current flow is maintained through a portion of molten material which is maintained in intimate contact with and which wets a solid portion generally of the same or related material. A magnetic field across the molten material having a component at right angles to the current flow produces a reactive force approximately equal and of opposite direction to any unbalanced force such as gravity acting on the material under treatment, whether liquid or solid, within the magnetic field. Stabilizing forces which compensate for small variations in reactive force due to variations in current flow and/or magnetic field strength and for deviations from the correct values of these parameters include surface tension in the liquid phase and adherence between the liquid and solid phases. Other such influences are normally present and will be discussed.

It will be seen that the described processes are particularly applicable to the zone-melting processes, reference above cited, in which there is at all times a liquid phase in contact with a solid phase of material undergoing treatment. In zone-melting in which a molten zone is caused to travel through a solid body of material by means of a moving heat source sufficient to melt the material, the suspension apparatus of this invention is ordinarily mechanically connected with the heating apparatus so that the moving molten zone is at all times within the influence of the magnetic field. In zone-melting, as practiced in accordance with this specification, current flow is generally produced through substantially the entire body undergoing treatment by establishing a potential difference across the ends of the body.

Although the processes here described are of obvious value in the zone-melting field, especially as applied to semiconductive materials, these processes are useful in the processing of materials other than semiconductors and as applied to processes other than zone-melting. For example, the suspension processes are useful in the forma-

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tion of a homogeneous solid ingot from a body of sintered material where the sintered mass is progressively fed into a heating means. The suspension methods are also useful in powder metallurgy where a molten body of material may be suspended while fresh powdered material is added as feed. In the last example, the molten material may be withdrawn from the heating means and allowed to crystallize as it passes out of the influence of the electromagnetic suspension apparatus all at a rate approximately equal to the rate at which new feed material is added to the molten portion or the current flow and/or field intensity may be gradually increased so as to compensate for the additional weight of the feed material.

Other uses include the suspension of reactive, molten metal for the purpose of alloying while avoiding contamination from a container and the suspension of material where it is desirable to expose a maximum surface area as for the purpose of adding a gaseous addition agent, removing a volatile constituent by evaporation or reacting an impurity of the melt with an agent in the atmosphere. These and other species of the suspension methods will be described.

For the purpose of teaching the described invention, reference will be had to the following figures:

Fig. 1 is a schematic diagram of the vector forces responsible for the suspension of material in accordance with the teaching of this invention;

Figs. 2A and 2B are schematic front and end views of a zone-melting apparatus in which the molten zone is prevented from contacting the container wall by virtue of the suspension method of this invention;

Figs. 3A, 3B and 3C are plan views of magnet faces shaped in such manner as to improve the uniformity of the magnetic field suitable for the practice of this invention;

Figs. 4A, 4B and 4C are diagrammatic views of magnet faces, the use of which results in non-uniform magnetic fields of decreasing horizontal component of field intensity in an upward direction which, when used in the practice of this invention, has a further stabilizing effect on the vertical component of the suspending reactive force;

Figs. 5A and 5B are schematic front and end elevation views of suspension apparatus adapted to the growing of homogeneous solid material from a sintered charge;

Fig. 6 is a front elevation view of suspension apparatus for use in crystallizing powdered or granular charge material;

Figs. 7A and 7B are schematic elevation and end views of suspension apparatus for use in a simple melting operation; and

Fig. 8 is a diagrammatic plan view depicting a particularly stable species of suspension treatment whereby a bulging body of liquid material is suspended within a non-uniform magnetic field.

Referring again to Fig. 1, a portion of a body 1 of material under treatment is indicated. A heating source, not shown, produces molten region 2, adjacent and contiguous with solid portions 3 and 4. Maintaining a voltage potential across the portion of body 1 shown results in a current flow here depicted as a vector 5 along the longitudinal axis of body 1. Vector 6 displaced 90 degrees in a counter-clockwise direction from vector 5 as viewed from the top represents a magnetic field component at right angles to and on a horizontal plane with body 1. The reactive force chiefly responsible for the suspension of molten region 2 is depicted as vector 7 shown displaced 90 degrees from vectors 5 and 6 and consequently manifesting itself in a vertical direction upwards.

In practicing the invention, the passage of direct cur-

rent **I** through the horizontal ingot **1** produces vector **5**. Vector **6** is produced by a horizontal magnetic field resulting, for example, from the placement of two pole faces of opposite polarity, one on either side of ingot **1**. By the application of heat from any conventional heating means, such as an arc, a resistance heater, or a high frequency inductive heater, a molten zone **2** is formed in ingot **1** within a magnetic field of magnetic intensity **H**. The interaction of current **I**, shown as vector **5**, and magnetic field of intensity **H**, shown as vector **6**, produces a reactive force **F**, shown as vector **7**, which, in this example, is directed vertically upward. The magnitude of force **F** is given by the well-known relation:

$$F = \frac{HI}{10} \text{ dynes} \quad (1)$$

where

H is magnetic intensity in units of oersteds,

I is the current passing through the material within the field in units of amperes, and

l is the length of the conductor within the magnetic field in units of centimeters.

An equation specifying the product **HI** needed to support a molten zone against gravity may be developed as follows: **F**, the suspending reactive force, is that force required to support a mass equal to that in the molten zone. Letting **W** equal the mass of the molten zone and **g** equal the acceleration of gravity, which is numerically equal to 980.6 centimeters per second squared, then:

$$F = Wg = \rho A l g$$

where

ρ = density in grams per cubic centimeter

A = cross-sectional area of ingot in square centimeters

Eliminating **l** by combining the above with Equation 1:

$$HI = 10 \rho A g = 9806 \rho A \quad (2)$$

For a cross-section of one square centimeter Equation 2 may be further simplified:

$$HI \cong 10^4 \rho \quad (2A)$$

It should be noted that in the above development, a uniform molten region cross-section and a uniform magnetic field intensity over the length of the molten region are assumed although, as will be shown, these conditions are not required for processing in accordance with the invention.

An illustrative example indicating the magnitudes of current and field intensity required will now be given. The material chosen is silicon. It is considered that the choice of silicon as an illustrative example is apt, since it is a semiconductive material which possesses many characteristics desirable in the design of semiconductive devices such as transistors and, further, since much difficulty has been had from contamination of the molten material by impurities contained in the retaining crucible.

Illustrative Example 1

Assuming a magnetic field intensity **H** of 2500 oersteds the current flow **I** required to suspend a molten zone of circular cross-sectional area of 6.45 square centimeters may be computed as follows. The density of silicon is 2.33 grams per cubic centimeter. From Equation 2:

$$I = \frac{9806 \rho A}{H}$$

Substituting the values given:

$$I = \frac{(9806)(2.33)(6.45)}{2500} = 59.0 \text{ amperes}$$

For the purposes of this example, it is assumed that the field is uniform and on a horizontal plane and that the current plane is at an angle of 90 degrees to the field also on a horizontal plane. Other stabilizing forces to be

discussed which permit some latitude in the magnitude of the reactive force are not considered. Assuming the resistivity of silicon to be 0.05 ohm-centimeter and the mean length of the ingot between electrode clamps to be 30 centimeters, the voltage required to pass the suspending current in this example is about 14 volts.

It should be noted that the resistivity value of interest is not that at room temperature but at the operating temperature of the material undergoing treatment. For semiconductive materials, the resistivity drops sharply as the temperature of the material is increased. If the electrodes are widely spaced so that they encompass a substantial amount of relatively cool material, it may be helpful to use auxiliary heaters around the solid portions of the material between electrodes.

The decrease in resistivity of silicon at a raised temperature is illustrative of the relationship for semiconductive materials. For silicon of a very high purity, the electrical resistivity at room temperature may be 100 ohm-centimeters or higher. However, the temperature of the molten region is at 1400° C. or higher so that some heating of the adjacent solid portions results. If it be assumed that a mean temperature of 500° C. in the solid ingot portions is maintained, the corresponding resistivity is about 0.5 ohm-centimeter. In illustrative Example 1, assuming a resistivity of 0.5 ohm-centimeter, a voltage potential of 140 volts is required to maintain a current flow of 59.0 amperes.

It is seen that the suspending force **F** is dependent only on the product **HI** and not on either **H** or **I** alone. For this reason a choice of values of **H** and **I** for a given suspension method is dictated by secondary considerations, such as economy, I^2R heating.

The most common limitation in the selection of parameters in the suspension method is the maximum available magnetic field intensity practically available. This is of special importance where the material undergoing treatment has a high resistivity so limiting practical current flow and/or introducing heat dissipation problems. Further, since the magnetic field may be supplied by permanent magnets involving no operating expense, and since increasing current flow increases operating expense, it may generally be desirable to operate at relatively high magnetic field flux and relatively low current flow. Exceptions to this suggest themselves: For example, in high resistivity materials in which the electrical resistivity decreases at elevated temperature, high current flow, and consequent I^2R heating may be desirable.

Values of magnetic intensity of 10,000 oersteds and greater are readily attainable between the pole faces of standard, commercially available, permanent magnets or electromagnets of moderate size in a gap length of one inch. Magnitudes of this value are obtainable over gap lengths of two inches and greater by resorting, however, to magnets which are larger than conventional size.

Frequently, it is desirable to achieve greater uniformity of magnetic intensity **H** longitudinally along the ingot. This is commonly done by increasing the pole face area thereby lowering the mean value of magnetic intensity. In such instances where some sacrifice in magnetic intensity is offset by the desirability of maintaining a uniform field, an expected range of field intensity may be of the order of from about 1,000 to about 5,000 oersteds.

The minimum value of usable magnetic intensity **H** depends on the optimum value of current **I**. This optimum value in turn depends upon such factors as the value of voltage available and the maximum permissible I^2R heating which latter may, in some instances, be limited by the energy required to melt the substance. It is noted here that large I^2R heat may be desirable, either for the purpose of decreasing the resistivity and hence allowing greater current flow, or for the purpose of minimizing or eliminating the required heater energy.

Further, as indicated, it is sometimes desirable to dissipate some of the I^2R heat through a cooling means and so maintain a value of current which would otherwise be sufficient to melt the material. From a mechanical standpoint, the use of high current and consequently smaller magnets or greater air gap may simplify the design of heating means for maintaining the material molten. A further effect, which may be deleterious, results from the use of high current flow in zone-melting in that the high I^2R heat decreases the temperature gradient of the trailing zone interface and consequently may result in less control of zone-length and, therefore, of composition.

A secondary limitation on the maximum value of current flow I of importance where the cross-sectional area of the ingot is small, is known as "pinch effect." This action tends to cause the molten region to neck down and separate under the action of the magnetic field of the current flow I . In general, any unintended influence tending to restrict or enlarge any portion of the suspended molten region is to be avoided since it will unbalance the equilibrium between suspended mass and suspending force F . As will be discussed, such variations, where desirable, may be maintained under equilibrium conditions by appropriate field flux gradients. Factors to be considered in determining optimum suspended region dimensions will be presented.

A typical zone-melting suspended zone installation is shown in Figs. 2A and 2B. The charge 10, for example, about one-half inch in diameter is enclosed coaxially in a quartz tube 11 of about a one inch bore which in turn is sealed to end blocks 12 and 13 by means of gaskets 14 and 15. End blocks 12 and 13 are mechanically supported by vertical members 16 and 17 which, in turn, are shown attached to movable supporting member 18. Gas inlet 19 and gas outlet 20 are embedded in end blocks 12 and 13 and make possible the maintenance of a desired atmosphere within quartz tube 11. During operation, the gas inlet 19 and outlet 20 permit filling the quartz tube 11 with a stationary or moving atmosphere of inert gas or of any other desirable gas intended to have some desired effect on the composition of the material under treatment. Also inlet 19 and outlet 20 may be used to permit the admission of a cooling or heating fluid. If it is desired to operate in an evacuated chamber, either inlet 19 or outlet 20 may be sealed and the vacuum pump attached to the remaining passage.

Suspending current I through body 10 is introduced by means of electrodes 21 and 22 which may be attached directly to body 10 as shown or which may be attached to end sections 12 and 13 providing these members are conducting. In general, the voltage applied across electrodes 21 and 22 is direct current supplied, for example, from a battery source or a rectified alternating-current source. It will be seen, however, that under certain circumstances, the voltage potential applied across electrodes 21 and 22 may be alternating as, for example, where the magnetic field is produced electromagnetically by an in-phase current. Further, stabilizing forces normally present during processing make permissible a suspending current I having an alternating-current component as may be produced by an unfiltered rectifier.

In operation, a mechanism, such as carriage 18, makes possible the longitudinal movement of body 10 between poles 23 and 24 which may, for example, be the north and south poles of an Alnico magnet or of two Alnico bar magnets of equal strength. Such an Alnico magnet may be in the shape of a large horseshoe of a weight of about 65 pounds. In the apparatus shown, magnet faces 23 and 24 may be pole faces of two separate bar magnets of dimensions of, for example, 3" x 3" x 9" mounted on a frame not shown, with their spacing adjustable so as to vary field intensity H .

Since the temperature of the magnetic pole faces may in operation rise to a temperature which is harmful to

the magnetic properties of the magnet or magnets, provision for passing a coolant over the pole faces is shown. In Fig. 2A coolant may be passed through hollow sections 25 and 26. The inner walls of sections 25 and 26 may be formed by the magnet faces themselves or may be distinct. In either structure a heat sink is interposed between the heating means 28 and the pole faces 23 and 24.

Molten zone 27 is formed within body 10 by heat interchange from heating source 28 which may be a torch, a resistance coil, a high frequency induction coil, or any other conventional heating means. In accordance with the zone-melting art, such heating means may include an inner ring of graphite, molybdenum or other high melting material which may assist in controlling the shape and location of either or both solid-liquid interfaces 29 and 30. It is understood that in the event the heating source 28 is a high frequency induction coil, a certain amount of inductive agitation occurs within molten zone 27. This may be desirable for stirring purposes, or may be undesirable in that it may tend to unbalance the equilibrium between the forces resulting in the suspension of the zone. It has been found, however, that a frequency source of the order of 5 megacycles produces only minor agitation and is suitable, although lesser frequencies of as low as the order of 450 kilocycles are usable.

It is understood that the depicted apparatus may be used for any of the zone-melting processes known to the art. These include "zone-leveling" in which the chief purpose is to produce a uniform distribution of a solute or solutes along a substantial length of the ingot or for "zone-refining" in which the chief purpose is to concentrate a solute or solutes in some desired location of the ingot. Zone-refining generally consists of repeated zone passes from near one end to near the other end of the ingot at a rate of about $\frac{1}{4}$ inch per minute to about $\frac{1}{50}$ inch per minute. Details on zone-length, number of passes, ingot length, travel rate, effect of the distribution coefficients of the components of the system, etc., may be found in the publication "Transactions of the American Institute of Mining and Metallurgical Engineers," volume 194, pages 747 through 753, 1952.

The entire tube assembly consisting of ingot 10, quartz tube 11 and end blocks 12 and 13, together with provisions for maintaining electrical current flow and a desired atmosphere within tube 11, may be rotated about its own axis to achieve improved uniformity of molten zone shape and heating. In certain instances, it may be desirable to rotate either end block 12 or 13 while maintaining the other in fixed position for this purpose and also to produce some stirring within molten zone 27.

It has been noted that the I^2R heating effect of the suspending current I through the ingot serves to heat the ingot to some extent. It will be shown that in some instances, this heating effect is sufficient to melt the material. In general, if the metal is a good electrical conductor such as, for example, aluminum, iron, tin, nickel, platinum and titanium, the current usually is insufficient to melt the ingot by itself. Nevertheless, the suspension current in such a situation takes some of the load off the zone heater.

It has been noted that alternating suspension current and reversing field flux if in-phase, neither having a direct or constant component, may be used in suspending a charge. If H and I are in-phase, the resultant reactive force F will be exerted in a constant direction although its value will fluctuate between zero and a maximum value at twice the frequency of the current and field. For this reason, it follows that where it is necessary or desirable to use in-phase alternating suspension currents and fields, the frequency should be sufficiently high so that the inertia of the system will prevent collapse. For this purpose, alternating-current suspension currents and reversing fields of a frequency of 60 cycles per second

have been found suitable, although higher frequencies are, in general, to be preferred.

Although all of the descriptive matter relating to the suspension method here under consideration has been in terms of a horizontal ingot and horizontal suspending currents and magnetic fields, it is to be understood that the described process is not so limited. The only general requirement is that the reactive force resulting from the interaction of the suspending current and magnetic field have a component which, together with the other stabilizing forces which are manifested, is sufficient to compensate for any force or forces tending to move the material to be suspended. Where the only force tending to move the material is due to gravity, the reactive force must have a vertical component in an upward direction and should not have a component in any other direction of magnitude sufficient to overcome any stabilizing forces such as surface tension tending to prevent motion in such direction.

Even where the force to be overcome is due solely to gravity, there are special circumstances under which it is desirable to use an ingot which is inclined from the horizontal. For example, during repeated passes in a zone-refiner operating on a material in which there is a difference in density between the solid and liquid phase, passage of the zones results in a small amount of matter transport which is cumulative with each pass. In simple zone-melting this may be overcome by inclining the ingot from the horizontal at such an angle as to compensate for this matter flow. See "Journal of Metals," November 1953, page 1441. In suspension zone-melting as practiced in accordance with this invention, matter transport may effectively be nullified in a similar manner, providing there are sufficient stabilizing factors which prevent motion of the suspended matter due to the non-vertical component of the reactive force. It may also be nullified by pulling or pushing one or both solid portions of the ingot to maintain the cross-section of the molten zone constant.

Uniformity of magnetic field flux through the zone volume is desirable although the restoring forces of surface tension and adherence between the liquid and solid phases at the interface may compensate for variations in flux. For ingots of the order of 1 or 2 centimeters in diameter or less, variations in flux of the order of 20 percent are tolerable assuming a surface tension of the order of 500 dynes per centimeter or more which is to be expected. As the ratio of mass to surface area of the suspended zone increases, the maximum permissible deviation from uniformity of field flux decreases.

Figs. 3A, 3B and 3C illustrate three simple mechanical expedients for obtaining a more nearly uniform field from given pole faces. It is recognized that for flat parallel pole faces, lying opposite one another, the field intensity is at a maximum between the centers of the faces providing the composition of the pieces is approximately constant across the faces. In each of the examples of Figs. 3A, 3B and 3C, deviation is made from parallel faces to configurations in which the air gap is decreased horizontally toward the extremities of the faces in such manner as to increase the lines of force in that direction and compensate for the effect above mentioned.

In Fig. 3A the faces of the pole pieces 40 and 41 are ground or otherwise shaped so as to produce vertical V-grooves in faces 42 and 43.

In Fig. 3B the faces of pole pieces 50 and 51 are shaped so as to produce the inwardly curved faces 52 and 53 thereby producing an air gap decreasing in length from the center to the horizontal extremities of the face.

Fig. 3C depicts pole pieces 60 and 61 cut back at surfaces 62 and 63 and leaving vertical ridges 64 and 65, thereby again resulting in a decreased air gap at the extremities of the faces and producing a more nearly uniform field intensity across the magnet faces.

Except where stated to the contrary, the remainder of

this specification is in terms of suspension methods in which the only unbalanced force to be opposed is due to gravity. It is to be further understood that references made to suspension current I and field intensity H are references made only to the components of these quantities which lie on a horizontal plane with I at a clockwise angle of 90 degrees to H as viewed in a downward direction. There may be other components of these values so that reactive force F opposing the gravitational force is only a component of the total reactive force. It is assumed that components of the total reactive force in a direction other than vertically upward are effectively negated by surface tension and other stabilizing influences.

Although, in general, a uniform field in a vertical direction normal to the common axis of the magnet faces is indicated, where it is apparent that instabilities exist in the system which tend to cause a change in the vertical position of the suspended material, an additional stabilizing influence may be introduced by resorting to the use of a graded magnetic field. Such a field should be arranged so that the horizontal component of magnetic intensity normal to the suspension current decreases in an upward direction in the region occupied by the suspended material. If, in such a field, instability caused by variation in suspending current, in magnetic intensity or in mass suspended tends to cause the suspended matter to drop, it will enter a portion of the field having a greater field intensity than its original position. The suspended matter will continue to drop until the increased field intensity increases the reactive force sufficiently to counteract the increased downward force. If, on the other hand, the unbalance is such as to produce a reactive force F which is greater than that required to counteract the effect of gravity on the suspended matter, the resultant upward component of F will cause the suspended matter to rise until the decreasing field intensity again brings the forces into equilibrium.

A graded field of the type described is always present to some extent in the field produced by flat parallel magnet pole faces. That is, in a field produced by such faces, the field strength is usually greatest along the line between the centers of the faces, assuming uniform magnet composition and no random lines of force, and diminishes with distance away from this line parallel to the faces. Therefore, if the suspended matter is so arranged as to be suspended above the center of the magnet faces, a dropping zone will put it under the influence of a field of increasing intensity while a rising zone will have the effect of decreasing the reactive force F.

If it appears that the fluctuations in either suspending current or magnetic field flux are too great to be counteracted by the vertical flux gradient present above the center of flat parallel pole faces, the slope of the gradient may be further increased. Methods of increasing this gradient include the use of fields produced by duplex magnetic structures incorporating weaker magnets or greater air gaps or both, in an upward direction, non-parallel pole faces so arranged that the air gap increases in an upward direction or magnetic shunts so as to shunt out part of the magnetic flux lines above the center of the suspended matter. As in all the methods herein described, it is to be understood that the magnetic field may be produced either by permanent magnets or electromagnets or by a combination of the two.

Figs. 4A, 4B and 4C illustrate three methods of producing non-uniform fields having a sharply decreasing horizontal magnetic field intensity in an upward direction. In Fig. 4A, faces 70 and 71 of magnet pole pieces 72 and 73 are ground or otherwise shaped in such manner as to produce a gradually increasing air gap and a consequent decreasing field intensity in an upward direction. This is shown schematically by the spreading of magnetic flux lines 74 upward. A suspended molten zone 75 is shown in cross-section. It is noted that the field gradient

has the effect of horizontally elongating the cross-section of the zone.

In Fig. 4B, a steeply graded field is produced by inclining pole pieces 80 and 81 at an angle from the horizontal so as to cause pole faces 82 and 83 to be deposited in a V shape. As depicted, the angle of the faces 82 and 83 is approximately 90 degrees. The increasing distance upwardly between lines of force 84 indicates a decreasing field intensity in an upward direction. The suspended zone is shown as an oval 86 which is the shape it acquires in a graded field.

The apparatus of Fig. 4C makes use of pole pieces 90 and 91 having flat parallel faces 92 and 93 and shows the use of a shunt 94 which may be composed of iron or of any other ferromagnetic material. Shunt 94, in the form of an inverted U or semicircle, is disposed with the open ends of the U at about the level of the top of the suspended zone 95. Shunt 94, in presenting less magnetic reluctance to lines of force 96, decreases the field intensity H within the ends of the U and above zone 95.

In addition to providing stability of a suspended zone against vertical motion caused, for example, by mechanical vibration, or by variation in suspending current or field intensity H, the fields shown in Figs. 4A, 4B and 4C tend to flatten the zones. For this reason, a zone which is produced from a rod of circular cross-section within such a graded field tends to assume a shape such as that of 75 of Fig. 4A or 86 of Fig. 4B. Although this may be disadvantageous where it is desired to retain a circular cross-section, it makes possible less distortion during melting and re-freezing where the ingot dimension of a horizontal plane is greater than that along the vertical axis as, for example, in the instance of a rectangular cross-section. It is apparent, therefore, that the design of the suspending apparatus may be such as to achieve the secondary purpose of distorting the cross-sectional shape of the molten zone or retaining the cross-sectional shape of the solid ingot to produce a final ingot of a desired shape. Variations in magnet face position and shape producing flux variations in the suspending field for the purpose of tailoring the final cross-section of the treated material will be apparent to those skilled in the art.

It should be noted that the fields of Figs. 4A, 4B and 4C, in increasing the vertical stability of the suspended zone, have the generally undesirable effect of introducing a type of instability not present in a uniform field. Since the lines of magnetic flux curve downward on either side of the ingot, as viewed in cross-section, there is a horizontal instability introduced so that if the suspended zone accidentally strays off center laterally, there is a tendency for it to continue in that direction. If the downward curvature is excessive, the loss in lateral stability introduced thereby may offset the gain in vertical stability obtained by grading the field.

In practice, on the basis of these considerations, a vertical gradation of field intensity H resulting from a decrease in a vertical direction through the center of the suspended zone of about 25 to 50 percent in a distance of one zone thickness in that direction is found suitable.

Thus far, the suspension methods of this invention have been described primarily in terms of the zone-melting processes. While it is true that electromagnetic suspension is of great value in such processes, the advantages of a suspended molten body are also valuable in other types of operations.

For electromagnetic suspension it is only necessary that the material to be suspended have sufficiently low resistivity at the operating temperature to permit the passage of adequate suspending current. It has been shown that the use of strong but commonly available magnetic structures makes necessary the passage of only minimal suspending currents. In fact, for reasonable

operating temperatures, it may be stated that the only materials upon which the processes herein may not operate are those having resistivities in excess of about 500 ohm-centimeters at the operating temperature. To operate in accordance with the teaching of this invention, it is also necessary that there be in suspension a body of liquid material which is in intimate contact with a body of solid material at at least one surface. Many processing operations in which these requirements are met are known in the metal industries and elsewhere.

Wherever, in the processing of a material which is not an insulator, it is necessary to maintain a body of liquid in contact with a body of solid, whether the liquid and solid be of the same chemical system or whether the materials be dissimilar so long as the one wets the other, the suspension processes may be used. These processes are valuable whenever, for reasons known to those skilled in the concerned arts, it is desirable to avoid contacting the liquid material with a crucible or other retaining wall. This may be desirable to avoid poisoning or other contamination from the crucible wall material, to avoid cracking resulting from expansion or contraction produced by melting or freezing the material under treatment, or to avoid crystalline imperfections in a material frozen from a melt otherwise introduced by a crucible wall.

A common objective in the metals industry is the formation of a solid ingot free of pores from a highly pure metal such as titanium which may be in the form of powder, tiny globules or small crystals. Sintering is sometimes used, but frequently results in porosity or in the entrapment of surface contaminants in the pores. Arc-melting in a water-cooled copper container has been used for metals like iron and titanium, but requires large power consumption and complex apparatus. Further, in arc-melting, failures in the copper containers are common.

One method of melting a powder and forming a solid ingot with the assistance of electromagnetic suspension may be carried out on the apparatus of Figs. 5A and 5B.

The apparatus of Figs. 5A and 5B resembles that of Fig. 2 in its general features, but differs from it in the nature of the charge. In this apparatus the charge 100 is a sintered compact in the form of a large rod. As in the apparatus of Fig. 2, charge 100 is contained within a tube 101 which may be constructed of quartz or other refractory material, which tube is sealed to end blocks 102 and 103 by means of gaskets 104 and 105. Gaskets 104 and 105 may be constructed of silicone rubber or other common gasket material which will withstand the operating temperatures and which will take up any imperfections between the bearing surfaces of tube 101 and end blocks 102 and 103. End blocks 102 and 103 are supported on vertical members 106 and 107 which in turn ride on a member such as carriage 108 which is free to move and so carry the charge progressively through a heating means. In operation, heat supplied by heating means 109 results in the formation of molten zone 110. Zone 109 is suspended by the reactive force resulting from the interaction of the magnetic field produced by magnetic pole pieces 111 and 112 and the suspension current I which is caused to pass through the charge by reason of an impressed voltage across electrodes 113 and 114. The remaining solid material 115 represents charge material such as that in charge 100 which has passed through the molten phase 110 and has frozen. As in the apparatus of Fig. 2, a gas inlet 116 and a gas outlet 117 are provided in the end blocks 102 and 103, respectively, so that a desirable atmospheric material, either moving or stagnant, may be maintained within quartz tube 101 or to permit evacuation of the chamber. In the event of evacuation, either inlet 116 or outlet 117 would ordinarily be sealed.

In the operation of the apparatus of Figs. 5A and 5B, charge 100 may be passed fairly rapidly, of the order of

1 to 10 inches per minute through heating source 109 which may be an induction heating coil. Where crystalline perfection or an absence of sharp compositional deviation is desirable the freezing rates are determined by reference to the zone-melting art.

Alternatively material may be melted and frozen by discrete zone lengths, for example by advancing the charge after each molten portion is produced or the temperature within the furnace may be lowered after each melting operation. Quasicontinuous operation is achieved by connecting successive sintered lengths.

Fig. 6 illustrates another melting technique in which a molten material is suspended by the reactive force resulting from the interaction of a magnetic field and a current which passes through the body. The apparatus depicted consists of end blocks 120 and 121, one of which, 121, is maintained in fixed position and the other of which, block 120, is attached to a movable member such as carriage 122. In operation, a starting ingot 123 is clamped at its extremities in blocks 120 and 121. A portion of ingot 123 is sealed within jacket 124 by fixed seal 131 and sliding seal 132 about which are disposed a heating means 125 and magnetic pole structure, not shown but resembling that of Fig. 5A.

To begin operation, a voltage is impressed across electrodes 126 and 127 contacting ingot 123, and molten zone 128 is formed by heat exchange with heating means 125. A desired atmosphere is maintained within jacket 124 by means of gas inlet 129 and gas outlet 130. Once molten zone 128 has been formed carriage 122 is moved in a left-hand direction so as to draw with it the solid portion of ingot 123 clamped in block 120. Simultaneously, powdered feed 133 is fed into inlet 134 at such a rate as to maintain the mass of material in molten zone 128 approximately constant.

A cavity or opening may be provided beneath molten zone 128 to prevent particles which do not melt into the zone from accumulating. Feed may also be in the form of a sintered rod or wire but in this event, precautions should be taken to maintain the suspending current constant. Adherence between the molten material of zone 128 and the solid material of the feed rod will help to stabilize the zone in position as against a variation in suspending current due to the introduction of a change in the electrical network.

If a granulated feed is to be used in the apparatus of Fig. 6, it may be desirable to use a rectangular or oval cross-sectional zone, thereby presenting a larger surface area to the feed. Magnet configurations which produce such zone shapes were described in connection with Figs. 4A, 4B and 4C.

The apparatus of Figs. 7A and 7B may be utilized for alloying or any process involving melting in which contact between the melt and the crucible wall is to be avoided. In such a process, a rod 140 of a material is clamped in blocks such as 141 and 142 and a current is passed through the rod 140 by means of an impressed voltage gradient across electrodes 143 and 144 making electrical contact with clamps 141 and 142, respectively. The portion of rod 140 between clamps 141 and 142 is melted by a heating means not shown, or solely by means of the I^2R heat generated by the passage of current through the rod 140. In either event, the molten region is suspended by the reactive force resulting from the interaction of the suspension current and the magnetic field produced by magnetic means not shown. If a constituent is to be added it may be introduced via a hopper such as member 134 of Fig. 6 or may be placed in contact with rod 140. In operation, the melt is formed and is then frozen in place. It will usually be desirable to cool clamps 141 and 142 to maintain the end portions of rod 140 solid.

By using clamps 141 and 142 containing means for automatically ejecting solid feed rods and means for

feeding new ones into them automatically, a series of such alloys may be prepared on a large scale basis.

An example of an alloy, in the production of which a technique such as that described is useful, is high purity nickel containing a few tenths percent of magnesium or aluminum. Alloys of such materials prepared in crucibles become contaminated by impurities in the containing walls or by the material of which the wall is constructed at the high melting temperatures of the alloys. Such alloys are of interest in the production of emitting elements for use in vacuum tubes.

On the basis of experimentation and of extended theory based thereon, generalizations may be made concerning the shape and sizes of suspended zones which are stable. Surface tension forces are the principal stabilizing forces maintaining the shape or position of a suspended zone. An imbalance between gravity and the suspension force, or a disturbance which temporarily alters the shape of the zone, results in an increase in surface area of the zone. Such an increase is opposed by the surface tension forces which tend to keep the surface area at a minimum.

A suspended zone is considered to be stable if a disturbance of an expected magnitude in the shape will be removed by the restoring action of surface tension forces. Other stabilizing influences which have been discussed will tend to introduce an additional safety factor. Adherence between the liquid and solid phases may result in the suspension of a molten zone, which, in accordance with these generalizations, is not stable when based on surface tension forces alone. At values of l/d greater than this value it is possible to contain the liquid volume in two separate spherical sections one at each interface, so that the total surface area is less than that for a cylinder.

A molten zone which is a circular cylinder of length l and diameter d at the solid-liquid interface is stable if l/d is less than about 3.14, or pi. A bulging or double convex zone corresponding approximately to a figure of revolution made by rotating an arc of a circle about the ingot axis is stable at values of l/d considerably greater than 3.14. A double concave zone in which the smallest cross-section diameter is no less than about one-half of the diameter at the liquid-solid interface is stable at values of l/d of up to about 1.5.

A given suspended zone shape is more stable at lower values of l/d . That is, the effect of the restoring forces of surface tension becomes greater as l/d becomes smaller.

There appears to be a maximum l which is attainable for a given substance, even at very small l/d ratios. It is estimated to be of the order of 3 inches for metals of moderate density and surface tension. A theoretical relationship for the value of the maximum stable zone length, $l_{\max}$, has been deduced, using simplifying assumptions, as follows:

$$l_{\max} = (\text{constant}) \times \left(\sqrt{\frac{\alpha}{\rho}} \right) \times (\text{shape factor}) \quad (3)$$

where

α = surface tension in dynes/centimeter
 ρ = density in grams/cubic centimeter
 $l_{\max}$ is in centimeters

The constant in Equation 3 is theoretically about 0.2 but in practice values of $l_{\max}$ have been obtained which correspond to a greater value for the constant.

Fig. 8 depicts top view of a pair of magnetic pole piece faces shaped in such manner as to suspend a bulging or double convex zone. Pole pieces 150 and 151 have wedge shaped faces 152 and 153, respectively. Since the air gap between pole faces 152 and 153 decreases in a horizontal direction from the extremities of the faces to the centers of the faces, the magnetic intensity of the resultant field is similarly graded so that the maximum intensity is in the line between the centers of the two faces

152 and 153 and falls off rapidly as the extremity of the faces is approached horizontally. Since the current passing through ingot 154 is of necessity constant over its length, it follows that the reactive force F is greater at the position in the ingot corresponding with the centers of the faces 152, 153. Such a force is capable of supporting a molten zone 155 which is of greater cross section in the portion of the zone corresponding with the region of greater magnetic flux.

For a given zone shape the restoring forces and hence the over-all stability will be greater for materials of greater surface tension and/or lesser density. The generalizations above set forth are based on a surface tension of about 500 dynes per centimeter. Actual surface tension values are generally somewhat higher than the cited value, illustrative values being 500 dynes per centimeter for tin, 1000 dynes per centimeter for copper, 1500 dynes per centimeter for iron and 700 dynes per centimeter for silicon. Other generalizations related to stability of a suspended zone follow:

The permissible variation of the horizontal component of the magnetic intensity H along the length of a suspended zone of uniform cross-section depends on the diameter and length of the zone and the density and surface tension of the material in the zone. Assuming the maximum value of the horizontal component of field intensity, H_x , to be at the center of the zone, the tolerable percentage change in H_x , ΔH_x , between the center and the ends is estimated as follows:

For:

$$\begin{aligned} d &\cong \frac{1}{2} \text{ inch} \\ l &\cong 1 \text{ inch} \\ \Delta H_x &\cong 15 \text{ percent} \end{aligned}$$

For:

$$\begin{aligned} d &\cong \text{approximately } 1 \text{ inch} \\ l &\cong \text{approximately } 1\frac{1}{2} \text{ inch} \\ \Delta H_x &\cong \text{approximately } 4 \text{ percent} \end{aligned}$$

For a double convex zone such as that described in connection with Fig. 8, the required value of H , at any position along the axis of the suspended zone, is directly proportional to the cross-sectional area of the zone at that position.

In general, vibrations in the apparatus should be avoided as these may affect zone shape and thereby produce instability. Reactive forces produced by the interaction of currents induced in the ingot by direct induction heating and those in the heating coil may agitate or distort the zone. To minimize this effect, the use of short zone-lengths having lengths of the order of less than twice the thickness are useful. Pulsations in the amplitude of the high frequency induction-heating power, at frequencies of up to about 360 cycles per second, should be avoided or at least minimized, as these may give rise to a resonant vibration of the zone which may lead in instability.

It has been found that induction heating has the tendency of producing a diminution in cross-sectional area at the center of a suspended zone. This effect can be substantially nullified by having oppositely-wound auxiliary turns at either end of the coil. A typical four-turn induction coil of a diameter of $2\frac{1}{2}$ inches and a spacing between centers of $\frac{3}{16}$ inch may to this end contain two oppositely-wound auxiliary turns spaced at $\frac{5}{16}$ inch between centers at either end.

Design criteria for the design of filter networks for use in filtering alternating-current supply currents are well known and are not here described. Reference to the generalizations set forth above indicating the percentage of stability permissible under certain operating conditions plus a knowledge of the required value of suspending current is sufficient information for the design of such a filter. In practice, it has been found that a filter network consisting of a 200-ampere rectifier with a 100,000-microfarad capacitance in parallel with the rectifier and a 150-microhenry inductance in series with

the load is sufficient to reduce a 50 percent ripple current in a 60 cycle-per-second alternating current to less than 5 percent of the direct-current value. A suspending current of 20 to 200 amperes has been obtained by use of such an arrangement.

A difficulty which is encountered in the suspension processes of this invention will be described. In general, the electrical resistivity of a given material varies from the solid to the liquid phase. For most metals, R , the ratio of electrical conductivity in the solid to that in the liquid is greater than 1. The ratio for tin is about 2.1. For certain metalloids such as antimony, bismuth and for semiconductors, R is less than 1.

If the process is one in which a solid body in a suspension field is melted through its cross-section, careful monitoring of either the suspension current or magnetic field will be required in order to maintain the reactive force constant. If the diameter or the density of the body of material under treatment is large, or if the surface tension of the liquid is low, the difficulty may be a serious one. However, this effect may be overcome as described.

It is apparent that, where R is defined as above, the suspension field may be maintained constant while smoothly changing the current I from a value of RI_1 in the solid ingot to an ultimate value of I_1 when the cross-section of the ingot is completely molten. Analytically, if a uniform length of ingot is melted in a strip which penetrates the cross-section of the ingot, the current I as a function of f , the fraction of the cross-section melted, may be given by the equation

$$I = I_1 [1 + R(1-f)] \quad (4)$$

in which I_1 indicates the calculated suspension current for the entirely melted zone.

Such an operation may be monitored electrically by measuring the resistance between two points on either side of the melting zone and using this information to program the value of I_1 . Alternately, field flux H may be monitored in accordance with the same relationship so that the product of I and H remains constant.

The above difficulty may be alleviated by supporting the melting portion at a fixed value of I and then removing the support once the molten zone is formed as, for example, by simply fixing the position of the support in relation to the ingot and by moving the zone away by usual zone-melting procedure.

Certain generalizations regarding the class of substances which may be processed in accordance with the instant invention are given below.

The conditions for magnetic zone suspension are given by the following equation:

$$HJ \cong 10^4 \rho \quad (5)$$

where

H = magnetic field intensity in oersteds
 J = current density in amperes per square centimeter
 ρ = density in grams per cubic centimeter

The following definition of J may be substituted in Equation 5

$$J = \frac{\sigma V}{l} \quad (6)$$

where

σ = mean electrical conductivity under actual conditions of operation in units of ohms⁻¹-centimeters⁻¹
 l = length of ingot between the current electrodes in centimeters
 V = applied voltage in volts

Combining equations 5 and 6:

$$\frac{\sigma}{\rho} = 10^4 \frac{l}{HV} \quad (7)$$

The parameters σ and ρ determine whether a substance

may be treated by the method of this invention. If reasonable maximum values of H and V and a reasonable minimum length l are substituted in Equation 7, a minimum value of σ/ρ is obtained. If a substance is to be treated successfully according to this invention, this minimum value must be equalled or exceeded.

For this purpose, a reasonable maximum value of field intensity is assumed to be 25,000 oersteds. Such an intensity may be obtained by use of Alnico permanent magnets or electromagnets in a gap length of the order of one inch. Stronger fields than that assumed are obtainable by use of air core solenoids using unusually large currents. The use of such magnets will result in a lower effective minimum value of the ratio σ/ρ and therefore permit suspension of a broader class of materials.

A reasonable maximum impressed voltage of 400 volts is assumed. It is recognized that this does not represent an ultimate limit.

A minimum length between electrodes L of 2.5 centimeters is assumed, although, in general for metals, an $L_{\min}$ of about 10 centimeters is preferred.

Substituting these values in Equation 6:

$$\frac{\sigma}{\rho_{\min}} \cong \frac{(10^4)(2.5)}{(25,000)(400)} \cong 0.0025$$

For metals, the maximum density ρ encountered is about 23 grams per cubic centimeter. Hence, the minimum conductivity σ is about $0.057 \text{ ohm}^{-1}\text{-centimeter}^{-1}$ for the densest metal. It is readily seen that all metals may be treated in accordance with this invention since values of σ for metals fall in the range of from about 10^3 to about $10^5 \text{ ohm}^{-1}\text{-centimeters}^{-1}$.

For semiconductors, the maximum value of ρ is about 10 grams per cubic centimeter. Hence, the minimum required value of σ is about $0.025 \text{ ohm}^{-1}\text{-centimeter}^{-1}$ or, expressed in terms of resistivity, the material under treatment should have a resistivity no greater than about 40 ohm-centimeters. Most semiconductors, especially when slightly impure, fulfill this requirement even on the basis of measurement at room temperature. Moreover, as was indicated above, the mean value of conductivity for a semiconductor under operating conditions approaching the melting point of the semiconductor may be orders of magnitude greater than the value at room temperature.

For salts and ionic solutions, the maximum densities encountered are about 4 grams per cubic centimeter so that the corresponding required minimum value of σ is equal to about $0.01 \text{ ohm}^{-1}\text{-centimeter}^{-1}$.

If σ for the material under treatment is low, on the basis of the assumptions made above, the power dissipated in the ingot may be sufficient to melt the entire ingot unless the solid portions are cooled. For example, the I^2R heat necessarily produced in an ingot 10 centimeters long, 1 square centimeter in cross-sectional area, having a value of σ equal to about $0.1 \text{ ohm}^{-1}\text{-centimeter}^{-1}$ at the minimum indicated current density of 4 amperes per square centimeter is equal to about 1600 watts. Such an amount of heat may be readily dissipated from an ingot having the indicated geometry by conventional cooling means.

Some of the advantages realized in processing according to the suspension methods here outlined follow:

(1) The suspension force is independent of zone-length as F is the lifting force per unit length of ingot. Actually, the entire portion of the ingot, solid or molten, which lies in the uniform field region is supported so that it makes little difference whether the material within the field is molten or solid.

(2) Surface tension forces act to maintain the position of the molten zone constant in line with the solid regions and help to prevent vibration due, for example, to small variations in field strength over the zone volume or small deviations of the field in a vertical direction. If the length of the molten zone is short, the surface ten-

sion forces will prevent a non-uniform field from distorting the shape of the ingot. Therefore, while the reactive force F is the chief influence in overcoming the effect of gravity on the suspended mass, surface tension forces provide a stabilizing influence which minimizes motion or distortion of the zone.

(3) The current per unit area throughout the cross-section and length of the zone is constant for zones having uniform cross-sectional area over the length. This effectively distributes the suspending force on an atomic basis throughout the zone volume. Therefore, providing the initial ingot is uniform in cross-section, the shape of the molten zone is not distorted by the suspending current. This is an advantage over levitation methods known to the art which rely on the use of induced eddy currents in the molten body.

(4) Another stabilizing influence compensating for vertical vibrations of the molten zone is introduced by the eddy current damping which results from the impressed field H . Motion of a suspended zone across a magnetic field results in induced eddy currents in a direction such that the fields of these currents oppose the motion.

(5) Control or adjustment of the suspending force is easily provided without affecting the heating power in the zone. Control means include varying the air gap by moving the pole pieces apart or together, varying the field intensity by charging the current in a direct-current electromagnet or in an auxiliary winding on a permanent magnet, or changing the value of suspending current. It is noted that where current flow I is substantial, a variation in I may be accompanied by changes in molten zone volume and hence in the rate of interface motion which may be undesirable in certain zone-melting operations and elsewhere.

It has been found that even a very thin oxide skin can greatly increase the stable zone-length. For example, in tin, a metal of relatively low surface tension, molten zones 2.4 inches long have been maintained in a $\frac{3}{16}$ inch diameter rod in air. A rather uniform H was used. H was furnished by an electromagnet with 4-inch diameter pole faces, $3\frac{1}{4}$ inches apart. H varied by less than 6 percent over the entire zone-length. Maximum stable lengths in a protective atmosphere for tin are about 1 inch.

Although the invention has been described in terms of suspension of material in an atmosphere of a gaseous substance or in a vacuum, the processes herein may be carried out in a heavier substance such as a liquid. Such a liquid may effectively reduce the density of the suspended zone and thereby make longer zone-lengths stable.

The suspension processes of this invention are, of course, applicable to the simultaneous suspension of two or more liquid regions. For example, in multiple zone, zone-refining all of the molten zones in the solid ingot may be suspended. In such a process a single magnetic field may encompass two or more zones so that, in effect, the solid region or regions intermediate the molten zones are also suspended.

In the above description, the use of the term "zone" connotes not only the molten zone-melting processes, but is intended to encompass all suspended bodies of liquid material. Other applications of the suspension processes have been suggested and still others will be apparent.

What is claimed is:

1. In a process in which a molten region is caused to traverse at least a portion of a solid body by melting material of the solid body at a solid-liquid interface, and freezing molten material of the said molten region at a solid-liquid interface, the procedure comprising passing a current through a conducting circuit including a current source and a portion of the said molten region and maintaining a magnetic field across a portion of the said molten region, such that there is on a horizontal plane a field component and a current component in which the said

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field component is at an angle of 90 degrees to the said current component, measured in a counter-clockwise direction as viewed downward, such that the said molten region is at all times in contact with the said solid body so that liquid-solid contact with the said molten region is limited to that at the said interfaces.

2. The process of producing a crystalline body from a compact granular mass, comprising causing a solid-liquid interface to traverse a portion of the said mass by melting granular material at the said interface, and causing a second solid-liquid interface to progress in the same direction as the first solid-liquid interface by progressively freezing molten material at the second solid-liquid interface, the procedure comprising passing a current through a conducting circuit including a current source and a portion of the said molten region and maintaining a magnetic field across a portion of the said molten region, in which the said field component is at an angle of 90 degrees to the said current component, measured in a counter-clockwise direction as viewed downward, such that the said molten region is at all times in contact with the said solid body so that liquid-solid contact with the said molten region is limited to that at the said interfaces.

3. The process of crystallizing a feed material comprising establishing in a solid body a molten region, adding feed material to the said molten region, and freezing molten material at at least one solid-liquid interface between the said molten region and a portion of the said solid body at a rate such that the quantity of molten material in the said molten region remains approximately constant, the procedure comprising passing a current through a conducting circuit including a current source

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and a portion of the said molten region and maintaining a magnetic field across a portion of the said molten region, in which the said field component is at an angle of 90 degrees to the said current component, measured in a counter-clockwise direction as viewed downward, such that the said molten region is at all times in contact with the said solid body so that liquid-solid contact with the said molten region is limited to that at the said interfaces.

4. The process comprising first melting and subsequently freezing at least a portion of a body of material in which there is at all times that there is molten material present, a solid-liquid interface between the said molten material and a solid material which is wet by the said molten material, the procedure comprising passing a current through a conducting circuit including a current source and a portion of the said molten region and maintaining a magnetic field across a portion of the said molten region, in which the said field component is at an angle of 90 degrees to the said current component, measured in a counter-clockwise direction as viewed downward, such that the said molten region is at all times in contact with the said solid body so that liquid-solid contact with the said molten region is limited to that at the said interfaces.

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