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Falckenberg

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[45] **Apr. 8, 1975**

[54] **VERNEUIL APPARATUS FOR GROWING SPINEL-TYPE OXIDE MONOCRYSTALS**

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[73] Assignee: **Siemens Aktiengesellschaft**, Berlin & Munich, Germany

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[21] Appl. No.: **271,669**

Related U.S. Application Data

[63] Continuation-in-part of Ser. No. 124,125, March 15, 1971, abandoned.

[30] **Foreign Application Priority Data**

Mar. 24, 1970 Germany 2014203
Nov. 24, 1970 Germany 2057782

[52] **U.S. Cl.** ..... **23/273 V**; 23/301 SP
[51] **Int. Cl.** **B01j 17/24**
[58] **Field of Search** ..... 23/301 SP, 273 SP, 273 V

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[57] **ABSTRACT**

The method of producing spinel-type oxide monocrystals wherein the crystal being grown is subjected to a relatively large temperature gradient in its axial direction and a relatively small temperature gradient in its radial direction. The invention includes laterally deflecting hot flame gases immediately below the growth front of the crystal and establishing heat equilibrium around the crystal being grown so that upon cessation of the flame gases, no sudden temperature drop is experienced by the crystal and fracture thereof is avoided. The deflection is achieved by a deflection member composed of a material having thermal characteristics similar to sintered Al_2O_3.

2 Claims, 10 Drawing Figures

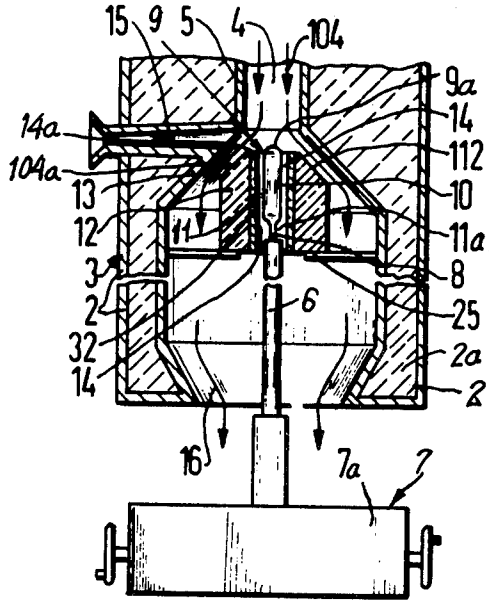


Fig. 1

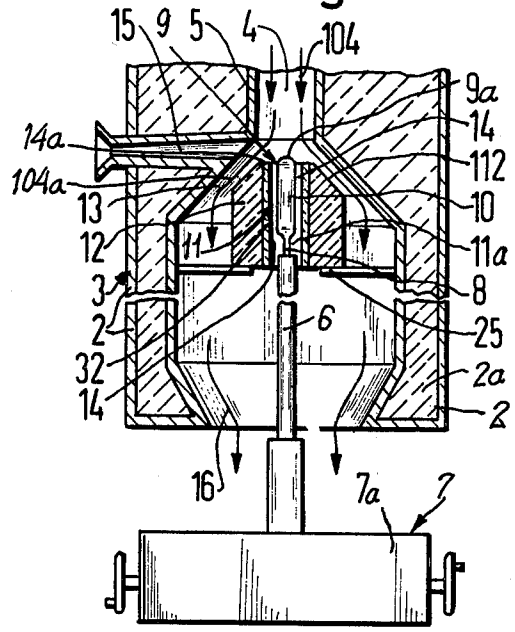


Fig. 2

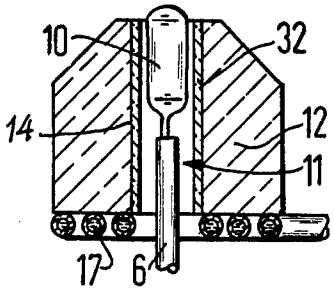


Fig. 3

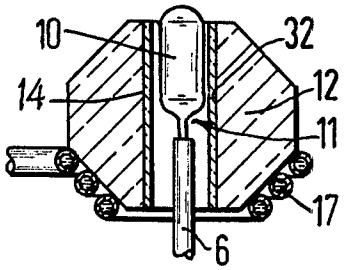


Fig. 4

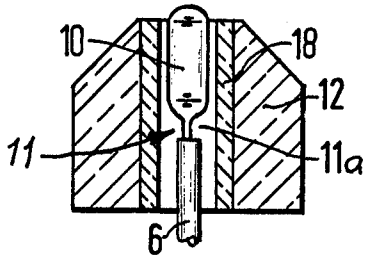
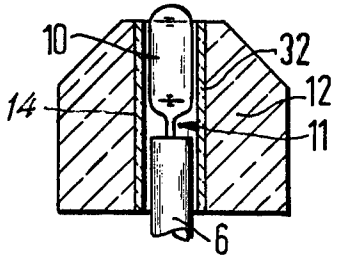


Fig. 5



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Fig. 6

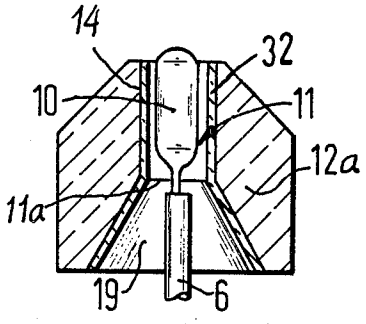


Fig. 7

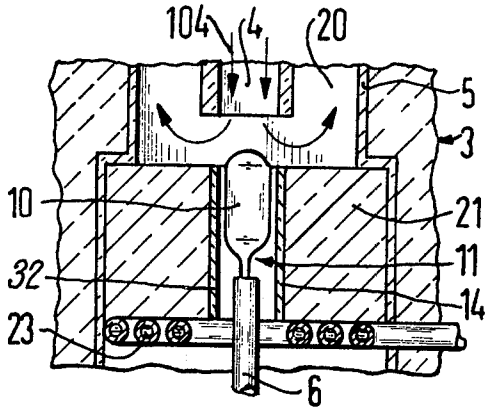


Fig. 8

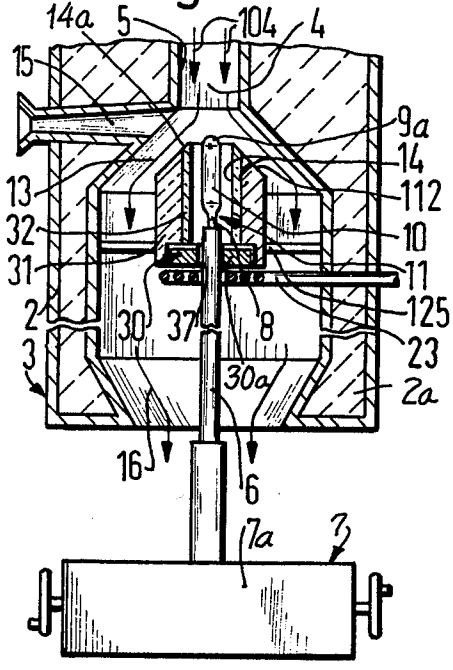


Fig. 9

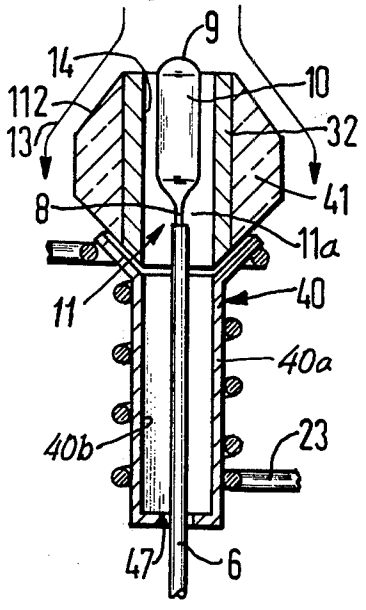
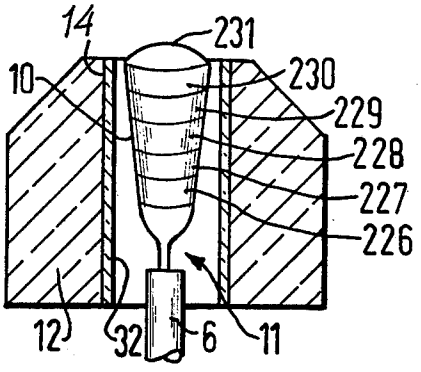


Fig.10



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VERNEUIL APPARATUS FOR GROWING SPINEL-TYPE OXIDE MONOCRYSTALS

CROSS-REFERENCE TO RELATED APPLICATIONS

This is a continuation-in-part application of U.S. Ser. No. 124,125, filed Mar. 15, 1971 and now abandoned. Attention is also directed to German Application No. 2,014,203, dated Oct. 14, 1971; Japanese Published Application 46-017149, published Oct. 30, 1971 as Japanese Patent Publication No. 3414/71; Netherlands Published Application 71.03970, Sept. 27, 1971; and French Pat. No. 71. 10 086, 068, issued Nov. 22, 1971, as well as to British Pat. No. 1,339,963, issued Apr. 3, 1974 and Canadian Pat. No. 942,638, issued on Feb. 26, 1974. all based on German patent applications No. P 20 14 203. 4, filed Mar. 24, 1970 and No. P 20 57 782. 6, filed Nov. 4, 1970; the priority of said German applications being claimed herein. All of the above disclosures are incorporated herein by reference. In addition reference is made to British Pat. No. 1,339,963 issued Apr. 4, 1974 and Canadian Pat. No. 942,638 issued Feb. 26, 1974.

BACKGROUND OF THE INVENTION

1. Field of the Invention

The invention relates to the production of spinel-type oxide monocrystals and somewhat more particularly to a method and apparatus for production of oxide monocrystals and/or mixtures of oxide monocrystals are characterized by low drift, low tension and high melting point characteristics.

2. Prior Art

One effective means of insulating integrated circuits comprises utilizing thin epitaxially separated silicon layers on insulating monocrystal substrate discs. Known materials for such substrate discs include sapphires and spinel crystals. Spinel crystals are preferred for a number of reasons, including the fact that spinels have the same cubic lattice symmetry as silicon and the fact that spinels are softer than sapphires and are, accordingly, more easily workable.

Spinels and/or spinel-type crystals are generally defined as the oxide and/or oxides of bi- and tri-valent metals of the formula $M^{II}O \cdot M_2^{III}O_3$ wherein $M^{II}$is, for example, Mg, Zn, Mn, Fe, Ni, Co or Cd and M^{III} is, for example, Al, Co, Fe, Cr or Ga. Spinels are generally synthesized in accordance with the Verneuil process. Presently, it is difficult to produce spinel crystals that are free from drift and grid flaw characteristics, i.e., dislocations and lattice defects. It is, of course, highly desirable to produce crystals which are free or substantially free of mechanical tensions which cause dislocations and lattice defects since any defects in the substrate material are transmitted and multiplied during epitaxial deposition on the substrate onto the ultimate semiconductor component and materially detract from the electrical properties thereof.

During the production of spinels in accordance with the Verneuil process, finely dispersed metal oxide powders are brought into contact with a heated and/or molten ridge of a seed crystal positioned in a hot zone. This seed crystal is moved out of the hot zone at a controlled rate adjusted to the melting rate of the oxide powders so that the molten powders cool and crystallize on the exposed ridge of the seed crystal.

Accordingly, it is an object of the invention to provide a method and apparatus for the production of oxide monocrystals having low tension and being substantially free from dislocations and lattice defects and particularly for the production of spinel monocrystals (for example, Mg-Al spinels) having low tension characteristics and stoichiometric or non-stoichiometric compositions, such as utilized for epitaxial coating with semiconductor materials.

SUMMARY OF THE INVENTION

The invention provides a method and apparatus for producing oxide and mixed oxide monocrystals of the spinel-type having few dislocations and low tension characteristics in accordance with a modified Verneuil process wherein the crystal being grown is subjected to a relatively large temperature gradient in its axial direction and a relatively small temperature gradient in its radial direction. Essentially, the invention deflects hot flame gases immediately below the growth front of the crystal so that only this small portion of the crystal is subjected to the direct action of the hot flame gases and establishing heat-equilibrium conditions around the remaining portion of the crystal so that upon cessation of the flame gases no sudden temperature change is experienced by the crystal and fracture is avoided. The temperature gradients and equilibrium conditions are generated by a lateral deflection member composed of a material having thermal characteristics similar to sintered Al_2O_3.

In its method embodiments, the invention comprises providing an operational environment for the growth of an oxide monocrystal and a suitable seed crystal therein, impinging hot flame gases and finely dispersed oxide powder on the growth front of such seed crystal, and substantially simultaneously laterally deflecting said flame gases away from the seed crystal immediately below the growth front thereof and establishing heat-equilibrium conditions about the growing crystal so that during impingement of said flame gases the crystal is subjected to a relatively large temperature gradient in the crystal axial direction and a relatively small temperature gradient in the crystal radial direction and during discontinuance of the impingement of such hot flame gases, the crystal is subjected to relatively constant ambient temperature conditions, free from sudden temperature drop and is then slowly cooled.

BRIEF DESCRIPTION OF THE DRAWINGS

FIG. **1** is an essentially diagrammatic elevational view, partially broken away, illustrating an apparatus embodiment useful in the practice of the invention;

FIGS. **2, 3, 4, 5, 6** and **7** are essentially diagrammatic elevational views of a portion of a modified apparatus useful in the practice of the invention;

FIGS. **8** and **9** are essentially diagrammatic elevational views, partially broken away, of a preferred apparatus embodiment useful in the practice of the invention; and

FIG. **10** is an essentially diagrammatic elevational view illustrating the growth of a spinel-type crystal in accordance with the principles of the invention.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

In its more general aspects, the invention provides

oxide and mixed oxide monocrystals of the spinel-type having low drift, low tension and high-melting point characteristics and methods of producing such monocrystals that avoid inducing mechanical stresses within the crystalline structure thereof. The monocrystals of the invention have stoichiometric or non-stoichiometric compositions; for example, stoichiometric crystals that require tempering prior to mechanical treatment, such as rubies; crystals which easily separate or demix, such as mullite crystals, such as ($Al_2O_3 \cdot SiO_2$); garnet crystals, such as $Y_3Al_5O_{12}$; spinel crystals useful in laser applications by appropriate additions, such as chromium and manganese; and/or jewel crystals. The invention is also useful in the production of ferrites and oxides having high melting points, for example, such as crystals composed of MgO or BeO.

The method embodiments of the invention generally comprise providing an operational environment for the growth of a select oxide monocrystal with an appropriate seed crystal therein, impinging a mixture of hot flame gases and a fine dispersion of the select oxide powder onto the growth front of the crystal, substantially simultaneously laterally deflecting the mixture of hot flame gases and oxide powder away from the seed crystal immediately below the growth front thereof and establishing heat-equilibrium conditions around the seed crystal, controllably moving the seed crystal downwardly relative to the flame gases at a select rate substantially equal to the growth rate of the monocrystal being produced for a period of time sufficient to achieve a desired size monocrystal, and then discontinuing the impingement of the hot flame gases and oxide powder so that during the impingement, the crystal is subjected to a relatively large temperature gradient in its axial direction and a relatively small temperature gradient in its radial direction and during discontinuance of the impingement, the crystal is subjected to a constant or relatively low decrease in temperature so that substantially little, if any, mechanical stress is introduced within the crystalline structure being formed.

In accordance with the known Verneuil technique for crystal growth, the growing or seed crystals are surrounded in their area of growth by hot flame gases so that the difference between the axial and radial temperature gradients on such crystals is quite small. For example, in the production of Mg-Al spinels having an excess of aluminum, the temperature gradient in the direction of growth is about 20° to 50° C./cm and such a small temperature gradient yields crystals having low mechanical stability and deficient electrical characteristics, as such crystals experience a very sudden drop in temperature or cooling when the hot flame gases are discontinued.

The apparent causes of the undesirable characteristics in crystals produced in accordance with the known Verneuil technique can be theoretically explained from a detailed consideration of the various facts involved. First, crystals grow with the desired characteristics on a seed crystal. When the hot flame is discontinued, the crystal jacket surface is suddenly cooled by several hundred degrees. This causes the jacket surface to contact and exert pressure onto the interior of the crystal. Such pressure causes a shifting in the interior structure of the crystal so as to form, with the initially present structure, granular limits or boundaries. Individual grains that are tipped against each other are thus readily produced. As a result, tension within the crystal is reduced. When the temperature inside of the crystal has dropped below the limit of plasticity (pressure-dependent), the above process ceases. The residual stresses can no longer be reduced and eventually lead to later fracturing of such crystal structures. It has been noted that rubies and spinels tend to fracture much less when subjected to sudden discontinuances of the hot flame than when subjected to gradual cooling. This result appears to be in accord with the above explanation. While slowly cooling, the entire crystal including its interior attains a given temperature within a relatively short time and below this temperature tension can no longer be reduced by plastic deformation of the crystal. With subsequent cooling, the tension becomes too great and leads to fracturing of the crystal structures. For materials that segregate or separate, it can be said that as a result of the small temperature gradient, the temperature zone in which separation, demixing, etc. can occur is proportioned to the time during which the layer is within this temperature zone and suffices to form segregations of the materials forming the crystal.

The invention provides a relatively large axial temperature gradient, preferably greater than about 200° C./cm and a very small radial temperature gradient, preferably smaller than about 10° C./cm on the growing crystal. Accordingly, during cooling the crystal jacket surface exerts only minimal, if any, pressure onto the interior of the crystal. In viewing a hypothetical picture of the atom layers in such a crystal perpendicular to the direction of growth, the topmost layer is subjected to the highest temperature and has the greatest heat expansion (i.e., lattice constant). The further one moves away from the growth front, the more the grid constant approaches the value it has at room temperatures. Accordingly, by cooling a crystal processed in accordance with the invention, there will be a uniform shrinkage of the crystal to the extent that first the bottom-most layer, then the central layer and finally the topmost layer will be at a normal lattice constant at room temperatures. This is purely an elastic process wherein no plastic deformation occurs. Thus, the invention has particular utility in the production of easily fracturable crystals. For example, stoichiometric spinels or crystals that require tempering prior to mechanical treatment, such as rubies; crystals which tend to separate or demix, such as mullite crystals, i.e., ($Al_2O_3 \cdot SiO_2$); crystals utilized for the production of garnets, such as $Y_3Al_5O_{12}$; spinel crystals capable of being utilized in lasers, as by the addition of chromium and magnesium; and/or crystals utilizable as jewels are all producible in accordance with the principles of the invention. Further, by the use of plasma burners (which allow higher temperatures to be attained), the invention provides a method of producing metal monocrystals having a high melting point, such as composed of niobium.

Since a growing crystal being processed in accordance with the principles of the invention attains room temperatures at areas thereof about 10 cm below its growth front, crystals of any desired length can be relatively easily grown. Utilizing conventional Verneuil burners, crystals having diameters of up to about 35 mm are readily attainable. The use of multiple burners during the production of large diameter crystals materially aid the process.

The crystals produced in accordance with the principles of the invention are substantially tension-free and generally do not require any further tempering. Such

crystals are substantially free of grain boundaries and the number of individual drifts are comparable to that observed in crucible-drawn crystals.

The principles of the invention are particularly appropriate for the production of stoichiometric and non-stoichiometric Mg-Al spinels for use as substrate discs in epitaxially applied semiconductor layers on integrated circuits. For example, in the production of Mg-Al spinel monocrystals having a stoichiometric composition, the invention provides that a select seed crystal is subjected to an axial temperature gradient of about 200° to 500° C. and a radial temperature gradient of less than about 10° C./cm at the growth front of the growing crystal while the remaining portions thereof are at heat-equilibrium conditions with their surroundings.

The principles of the invention are also useful in synthesizing rubies, such as utilized in lasers, particularly in the form of giant rubies. Another area wherein the principles of the invention are useful, is in the production of ferrites and oxides having high melting points, for example, such as MgO or BeO.

The apparatus embodiment of the invention generally comprises a crystal growth furnace means in an operational environment and a hot flame deflection means positioned within the furnace means so as to encompass a substantial portion of a seed crystal while exposing a select area of such crystal to impingement by a mixture of hot flame gases and oxide powder being fed into the furnace means so that molten oxide powder or droplets accumulate on the exposed area of the crystal and define a growth front. The hot flame gases and oxide powder is laterally deflected away from the seed crystal immediately below the growth front thereof so that the growing crystal is subjected to a relatively large temperature gradient in its axial direction and a relatively small temperature gradient in its redial direction while the remaining portions of the crystal are subjected to heat-equilibrium conditions with their surroundings, i.e., with the temperature of the deflection member. The deflection member is composed of a material having the thermal characteristics of sintered Al_2O_3 so as to withstand the hot flame gases and have sufficient heat capacity to prevent a sudden drop in the temperature of the crystal after discontinuance of the flame gases. The deflection member and the crystal remain at essentially identical temperatures in adjacent zones during and after impingement of such flame gases. Preferably, the deflection member is relatively massive for improved heat capacity characteristics.

In a preferred apparatus embodiment, the gas deflection member is provided with a centralized hollow passage so that the seed crystal can be positioned within the growth furnace through the hollow passage and have only the growth front thereof exposed to the impinging hot flame gases and oxide powders. In certain embodiments, seal means are provided around the centralized passage away from the growth front of the crystal so as to seal the inner area of the centralized passage, preventing entrance of the hot flame gases from entering the area and more readily attain heat-equilibrium conditions between the deflection member and the crystal. The seed crystal is suitably supported for select movement within the deflection member so that the seed crystal can be controllably moved in accordance with the growth process.

Referring now to the apparatus embodiments illustrated in the drawings, wherein like reference numerals denote like elements, a somewhat cylindrical crystal growth furnace means **3** is diagrammatically illustrated at FIG. **1.** The furnace means **3** is provided with a hollow inner chamber. Only the lower portion of the furnace means **3** is illustrated since the upper portion comprised of a powder feed system which can include a vibration box, a hammer means and/or other pulverizing and vibrating means; a gas burner and nozzle, appropriate feed lines, supply means providing hydrogen and oxygen and/or inert gas (all not illustrated) merely forms an operational environment (and not illustrated) merely forms an operational environment (and it will be so-referred to in the description and claims) for a crystal growth process and such operational environment is generally similar to the conventional Verneuil apparatus.

In one form of the apparatus embodiment, furnace means **3** is about 350 mm in height and jacketed and an aluminum panel **2** and appropriately filled with fire clay so as to have an outer diameter of about 250 mm. A gas supply inlet means **4** is suitably provided for feeding a gas-powder mixture into the interior of furnace means **3** as shown. The gas supply inlet means **4** has an inner diameter of about **35** mm and is provided with a layer **5** of a reflective non-contamination material, such as aluminum oxide, so as to prevent various impurities from commingling with the gas stream. A gas deflection means **12** is positioned in the centralized inner portion of the furnace means **3** and supported by appropriate spider-like bridge means **25,** which are anchored to the inner side walls of the furnace means **3.** The outer surfaces of the gas deflection means **12** and the inner walls of the furnace means **3** serve to define a lateral deflection chamber **13.** The hot gas stream or jet carrying a supply of oxide powder travels through the inlet means **4** and impinges on the growing crystal **9,** as generally indicated by arrows **104.** A crystal holding means **6,** as for example, composed of sintered aluminum, is positioned so as to protrude into the centralized interior portion of the furnace means **3** and into the centralized hollow passage **11** of the gas deflection means **12.** A support means **7,** such as a gear block member **7***a* is positioned below the furnace means **3** to movably support the crystal holding means **6.** The gear block member **7***a* allows the crystal **9** to be rotated and/or moved up and down as desired, either manually or by an appropriate motor means. The excess mixture of flame gases and oxide powder exit from the furnace means **3** through an outlet means **16.**

At the start of the process, a suitable seed or nucleus crystal **8** is positioned on the crystal holding means **6** and heated by the oxyhydrogen gas flame in an area above the gas deflection means **12.** Generally, the seed crystal **8** is positioned so as to be about 5 mm above the upper surface of the deflection means **12.** This causes a distance adjustment between the gas nozzle (not shown, but having a flame discharge of about **3** mm in diameter) and the growth front **9***a* of the spinel **10** being grown to be produced by the powder supply fed to the gas stream. This distance is about 9 cm. The actual growth area in the furnace means **3** is indicated by reference numeral **11***a* and is the area encompassed between the inner wall portion **14** of the deflection means **12** and the outer surface of the growing crystal **9.** In other words, the growth area **11**a is defined by the area

of the gas deflection means **12** which encompasses or envelops the crystal being grown.

The gas deflection means **12** is composed of a material having thermal characteristics adapted to the particular crystal being grown, i.e., having heat resistance and heat capacitance in accordance with the thermal characteristics of the crystal being grown. Since sintered aluminum oxide has the requisite thermal characteristics, the material forming the deflection means preferably has thermal characteristics similar to that of sintered aluminum oxide. For example, when a Mg-Al spinel is being produced, a gas deflection means composed of sintered Al_2O_3 is preferred. As shown, the deflection member is relatively massive for improved heat capacity. The function of the gas deflection means **12** is to deflect the hot flame gases that are impinging from the inlet means **4** onto the exposed area of crystal **9** and preferably to deflect such gases at the area immediately below and away from the growth front **9***a* thereof. This function is advantageously accomplished by selecting or forming the upper surfaces **112** of the gas deflection means **12** into cone-shaped or beveled surfaces, such as shown, so as to direct the flame gases into the lateral deflection channels **13** along a path generally indicated by curved arrows **104**a and away from the growth front **9***a*. The beveled or cone-shaped upper surfaces **112** also function to guide any excess oxide powder away from the deflection member **12.** During flame impingement the gas deflection means **12** is heated up and because of its thermal characteristics it provides heat-equilibrium conditions to the remaining portion of the crystal being grown after the discontinuance of the flame gases so as to minimize any thermal shock to the crystal being produced and thereby reduce the mechanical stresses therein.

Suggestions have been made to use a crystal holding means **6** shaped so that in the area below the seed crystal **8,** it is expanded or thickened in cross-section so that the flame gas flow resistance through the centralized passage **11** of the deflection means **12** is high relative to the resistance to gas flow in the lateral deflection channels **13.** Accordingly, as small a space as possible (about 1 mm tolerance) is provided between the holding means **6** and the inner wall **14** of the gas deflection means **12.** The holding means **6** is shaped so as to substantially fill the cross-section area of the centralized passage **11.** To the extent that a growing crystal expands, the crystal adapts itself to the space available. In this manner, encompassment of the growing crystal by the hot flame gases is avoided. The internal or centralized passage **11** within the gas deflection means **12** has a diameter of about 20 mm and can be provided with a suitable reflective layer along the inner walls **14,** for example, such as composed of Al_2O_3.

In order to avoid fusing a growing crystal **9** with the inner walls **14** of the gas deflection means **12,** the upper edges **14***a* of the deflection means **12** are slightly beveled and provided with smooth surfaces. During the growth process, a major portion of the supplied oxide powder impinges directly on the exposed crystal area. Proper adjustment of the distance between the growth front of the crystal and the upper edge of the growth area allows any excess powder to be carried away by the passing flame gases to the outlet means.

A viewing port **15,** having a diameter of about **10** mm is provided so as to provide a view of the growth front inside the furnace means **3.** The viewing port **15** preferably opens inwardly, much like a rifle sight, so that the growth front of the crystal can be fully viewed. In a modified embodiment, additional viewing ports are provided, each spaced about 90° from the other.

A specific apparatus useful in the practice of the invention and constructed in accordance with the illustration at FIG. **1** has the following dimensions:

Inner diameter of the centralized passage 11 of gas deflection means 12	20 mm
Width of lateral deflection channels 13	10 mm
Maximum diameter of gas deflection means 12	80 mm
Height of gas deflection means 12	70 mm
Angle of inclination of surfaces 112 of deflection means 12	45°

This form of apparatus provides an axial temperature gradient of about 200° to 500° C./cm and a radial temperature gradient of less than about 10° C./cm onto the crystal being grown. The growing rate of a crystal is about **1** to **2** cm/hour and a typically grown crystal has a diameter of about 18 mm. In order to maintain a constant diameter, a continuously small increase in the amount of oxygen being supplied is provided.

FIG. **2** illustrates a modified embodiment of the gas deflection means **12.** Cooling means **17,** such as water conduits or the like, are provided along the bottom surface of the deflector means **12** to further increase the axial temperature gradient on the spinel **10.**

FIG. **3** illustrates a further modified embodiment of the gas deflection means **12.** Cooling means **17** are provided along side portions of deflector means **12** to further increase the axial temperature gradient within the central passage **11** of the deflection means.

Further increase in the temperature gradient on the growing crystal in its axial direction is provided by layer **32** on the peripheral walls **14** of the central passage **11** of the deflection means **12.** A layer **32** is composed of a material having high heat conduction characteristics similar to that of sintered aluminum oxide. The deflection means **12** and the layer **32** absorbs sufficient heat from the hot flame gases during the impingement thereof onto deflection means **12** so that during discontinuance of the flame gases the layer **32** radiates sufficient heat to maintain the crystal in heat-equilibrium with its surroundings and thereby avoiding thermal shock and mechanical stress. Preferably, layer **32** is composed of densely sintered aluminum oxide, since this material has a relatively high heat conduction characteristic and has thermal characteristics similar to sintered aluminum oxide. For similar reasons, a suitable material having the thermal characteristics of sintered aluminum oxide, for example, such as densely sintered aluminum oxide, is utilized to form the crystal holding means **6.**

FIG. **4** illustrates a portion of an embodiment having a liner **18** provided along the peripheral surfaces of the central passage **11** of the gas deflection means **12.** The liner **18** is composed of a material having required thermal characteristics as outlined above and, for example, may comprise densely sintered aluminum oxide.

FIG. **5** illustrates a portion of another embodiment having a crystal holding means **6***a* composed of a material having thermal characteristics similar to densely sintered aluminum oxide.

FIG. **6** illustrates a portion of yet another embodiment having a gas deflection means **12***a* provided with an outwardly flared lower portion **19** and comprising

the lower portion of central passage **11** of the gas deflection means **12***a*. Such shaped lower portion **19** provides improved downward heat transfer away from the growing spinel **10.**

FIG. **7** illustrates a portion of a further embodiment having gas deflection channels **20** arranged so as to direct the flame gases upwardly and away from the growing spinel **10.** In this embodiment, a gas deflector means **21** is of a planar shape and not conically shaped as in the other embodiments discussed. As shown, gas deflection means **21** is sufficiently massive to fill a substantial portion of the interior of the furnace means **3** so as to form an improved heat reservoir. Cooling means **23** are operatively associated along the bottom portion of the deflection means **21** to cool the deflection means. It will be appreciated that such cooling means **23** can be provided along the side surfaces of the deflection means, as illustrated in FIG. **3.**

FIGS. **8** and **9** illustrate particularly preferred apparatus embodiments useful in the practice of the invention. In such apparatus, the gas deflection means (**31,** 41) is provided with a substantially gas impermeable seal means (**30, 40**). The seal means is positioned opposite and away from the growth front of the growing crystal and below the central passage **11** of the deflector means (**31, 41**). The seal means is provided with a relatively small aperture or passage (**37, 47**) to permit the crystal holding means **6** to pass therethrough and substantially prevent any hot flame gases from passing through the central passage **11.**

In the embodiment shown at FIG. **8,** the seal means **30** comprises a plate member **30***a* having a small passage or aperture **37** for admitting the crystal holding means **6.** The plate member **30***a* is positioned so as to seal the central passage **11** and particularly growth area **11***a* so that higher temperatures are attained around the spinel **10.** The plate member **30***a* is anchored, as by cementing, onto the gas deflection means **31** and preferably rests on support members **125,** which are somewhat similar to the bridge means **25** discussed at FIG. **1.**

In the embodiment illustrated at FIG. **9,** a gas deflection means **41** is provided with seal means **40** comprised of an elongated body or pot **40***a* having a passage **40***b* therein. Pot **40***a* is suitably attached to the lower surface of deflection means **41.** The passage **40***b* is of a sufficient axial dimension to allow the crystal holding means **6** and/or the seed crystal **8** to be lowered a distance sufficient to allow the entire growing spinel **10** to be accommodated in the central passage **11** of the deflection means **41** and in the passage **40***b* of the body **40***a*. A small aperture **47** is provided along the bottom surface of body **40**a to accommodate the crystal holding means **6.** Cooling means **23** (such as water conduits or the like) are provided along the outer surface portions of the body **40***a* and, if desired, can also be operationally associated with appropriate surfaces of the deflection means **41.** The remainder of the furnace means associated with the portion illustrated is similar to the furnace means **3** discussed in conjunction with FIGS. **1** and **8.**

The specific preferred form of the apparatus illustrated in FIGS. **8** and **9** have the following dimensions:

Inner diameter gas inlet means 4	28 mm
Inner diameter of central passage 11 of gas deflection means 31, 41	35 mm
Width of deflection channels 13	10 mm
Maximum diameter of gas deflection means 31, 41	80 mm
Height of deflection means 31, 41	70 mm
Angle of inclination of surfaces 112 of gas deflection means 31, 41	45°

It is generally preferred to provide inlet means **4** with a diameter smaller than the diameter of the central passage **11** of the gas deflection means (**12 12***a*, **21, 31** or **41**). In this manner, the central passage **11** is exposed to less flame gas and an increase in the powder yield is achieved.

The gas deflection means illustrated in the various Figures (i.e., **12, 12***a*, **21, 31** or **41**) can be provided with a liner **32** on the inner walls **14** of the central passage **11.** The liner **32** is composed of a material having thermal characteristics similar to densely sintered aluminum oxide and in the preferred embodiment is composed of densely sintered aluminum oxide.

An advantage of the apparatus illustrated at FIGS. **8** and **9** is that practically all ingress of hot flame gases into the area between the growing crystal and the deflection means is eliminated by the lower seal means. This area can thus be maintained sufficiently large to safely exclude any melting or fusing of the growing crystal onto the deflection means. Such apparatus has seal means (**30, 40**) with openings (**37, 47**) that are much smaller than the remaining space between the holding means **6** and the deflection means **12** illustrated at FIG. **5.**

Preferably, the necessarily remaining gap (**37, 47**) between the crystal holding means **6** and the seal means (**30, 40**) in the apparatus utilizing such a construction, is sealed with an elastic heat-resistant material, such as for example, rock wool and/or corundum wool.

Another advantage of the invention is that the temperature gradient in the furnace means is substantially independent of the length attained by a growing crystal.

An additional means of increasing the temperature gradient in the axial direction is to form the gas deflection means (**12, 31, 41,** etc.) of a material having relatively poor heat conduction characteristics (i.e., having a heat conductivity of less than about 0.5 K cal/mh °C.) and/or to form the portion of furnace means **3** below the deflection means from a material having relatively high heat conductive characteristics (i.e., having a heat conductivity of more than about 0.5 K cal/mh °C.). A further means of increasing the temperature gradient is to include appropriate heat exchange means, such as circulating cool water or streams of a gas along appropriate portions of the furnace means and/or the gas deflection means.

FIG. **10** illustrates a spinel crystal **10** during its growth process in accordance with the principles of the invention. The crystal **10** is positioned within a central passage **11** of a gas deflection means **12.** The sequential zones **226–231** represent the growth of individual growth zones of the spinel crystal **10** caused by increasing the lattice constants with increased temperatures.

Modifications and variations may be provided without departing from the spirit and scope of the novel concepts of the invention.

I claim as by invention:

1. An apparatus for producing oxide monocrystals comprising:

a crystal growing furnace means in an operational environment and having a hollow portion therein;

means feeding hot flame gases and oxide powder in a given direction into said hollow portion;

a relatively massive deflection means positioned within said hollow portion and in spaced registry from said means feeding hot flame gases and oxide powders,

said deflection means comprising a solid body composed of sintered aluminum oxide, said body having a centralized hollow passage of a given diameter, upper surfaces extending radially away from said passage for laterally deflecting hot flame gases away from said passage, and a width dimension greater than said diameter so as to retain heat within said body and function as a heat-reservoir for a crystal within said passage; and

a movable crystal holding means including a seed crystal, said holding means extending into said passage of the deflection means and aligned so that said seed crystal barely protrudes above the upper surface of said deflection means.

2. An apparatus as defined in claim **1** wherein said centralized hollow passage of the deflection means is provided with a heat-resistant gas-impermeable seal means positioned below the upper surfaces of said deflection means and in a movably working relation with the crystal holding means allowing said holding means to move relative to said seal means.

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