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CERAMIC SUPPORT PLATE

Abstract

Disclosed herein is a device for manufacturing a single crystal comprising: a furnace that includes a furnace wall; a crucible disposed on a first base plate within the furnace; where the first base plate has a porosity of 1 to 80 volume percent, based on a total volume of the first base plate; and where the first base plate does not comprise free flowing particles; an induction coil disposed inside the furnace wall and outside the crucible; and a refractory lining being disposed in an annulus between the furnace wall and the crucible.

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Background/Summary

BACKGROUND

[0001] This disclosure relates to a ceramic plate for supporting a crucible in a furnace. More specifically, this disclosure relates to a ceramic plate that is used for supporting a crucible that contains a melt from which high temperature materials are manufactured.

[0002] During the manufacturing of a crystal boule in a furnace, the crucible is heated by inductive heating using inductive coils that are located outside the outer tube. During this process, the raw materials in powder form are located in the crucible and are melted in a processing atmosphere (typically an inert gas) to prevent oxidation during the growth and cooling stages.

[0003] The crucible is typically manufactured from a noble metal. The crucible is supported by or in contact with refractory materials of any configuration. For example, the refractory materials may be ceramic-ceramic composite materials of any shape, such as cylinders, plates, sheets, fabrics, spheres, fragments, and the like. They may be solid or hollow beads of various surface areas. The chemical compositions of the refractory materials include ceramics that include, but are not limited to silica, alumina, magnesium oxide, calcium oxide, calcium silicates, hafnium oxide, zirconium oxide in any combination and form. Depending on the furnace design, refractory placement, the growth atmosphere selected, the composition of the crucible material and the composition of raw materials from which the single crystal is to be grown, oxidation of the crucible may occur. Points of contact between the refractory materials and the crucible results in the noble metals (used in the crucible) diffusing into the refractory materials. Local evaporation of the noble metal due to oxidation of the crucible may also occur. This produces a loss of the noble metal (which is expensive) and increases the costs of manufacturing the single crystals. It is therefore desirable to minimize the loss of metal from the crucible and to reduce the costs of manufacturing the single crystals.

SUMMARY

[0004] Disclosed herein is a device for manufacturing a single crystal comprising a furnace that includes a furnace wall; a crucible disposed on a first base plate within the furnace; where the first base plate has a porosity of 1 to 80 volume percent, based on a total volume of the first base plate; and where the first base plate does not comprise free flowing particles; an induction coil disposed inside the furnace wall and outside the crucible; and a refractory lining being disposed in an annulus between the furnace wall and the crucible.

[0005] Disclosed herein is a method of growing a high temperature material, comprising disposing a melt within a crucible in a furnace; where the furnace includes a furnace wall; an induction coil disposed in an annulus between the crucible and the furnace wall; where the crucible is disposed on a first base plate within the furnace; where the first base plate has a porosity of 1 to 80 volume percent, based on a total volume of the first base plate; and where the first base plate does not comprise free flowing particles; and drawing a boule from the melt within the crucible to grow the high temperature material.

Description

BRIEF DESCRIPTION OF THE FIGURES

[0006] FIG. **1** is a schematic depiction of an exemplary device that is used to produce a crystalline boule;

[0007] FIG. **2** is a schematic depiction of an exemplary monolithic base plate; and

[0008] FIG. **3** is a schematic depiction of an exemplary base plate that comprises 4 monolithic pieces.

DETAILED DESCRIPTION

[0009] Disclosed herein is a ceramic-ceramic composite base plate that is disposed beneath the crucible in a furnace. The base plate supports the crucible and isolates it from the ‘bubble grog ceramics' that can become imbedded in the crucible during a crystal growth heat cycle. At least one surface (e.g., an upper surface of the base plate) contacts the bottom outer surface of the crucible. The upper surface of the base plate comprises a plurality of ridges and channels, which alternate with each other. The upper surface of the ridges contacts the bottom surface of the crucible and establishes contact points between the two surfaces. Disclosed herein too is a device that uses the base plate during the manufacture of a single crystal boule.

[0010] The crystalline boules may be single crystal boules, with dopant activators to form a scintillating material, that may also contain co-dopants to enhance the scintillator performance characteristics and/or improve mechanical/physical properties. In an embodiment, the crystal boules may comprise high temperature materials that are not oxides crystals or oxide scintillators.

[0011] Examples of such scintillators are, but are not limited to: lutetium oxy-orthosilicates (LSO's), lutetium yttrium oxy-orthosilicates (LYSO's), gadolinium oxy-orthosilicates (GSO's), gadolinium aluminum gallium garnets (GAGG's), gadolinium-gallium-aluminum garnet (GGAG's), gadolinium-yttrium-gallium-aluminum garnet (GYGAG's), gadolinium-lutetium-gallium-aluminum garnet (GLuGAG's), gadolinium-scandium-gallium garnet (GSGG's), gadolinium-yttrium-aluminum garnet (GYAG's), gadolinium-scandium-aluminum garnet (GSAG's), gadolinium-gallium garnet (GGG's) or gadolinium-yttrium-scandium-aluminum garnet (GYSAG's).

[0012] FIG. **1** depicts device **100** that is used to produce a crystalline boule. The device **100** comprises a furnace **102** that contains cooling tubes **103** disposed in its walls. Alternatively, the cooling coils may be located outside the walls of the furnace. Fluid flowing through the cooling tubes **103** can be used to extract heat from the furnace. The furnace **102** is mounted on a base surface **104** and has a furnace cover **106** disposed on an end opposite the base surface **104**. The base surface **104** may contain channels that can be used to locate induction coils (not shown), coils for carrying a cooling fluids (not shown) or components that can facilitate mechanical movement (not shown). Disposed in the furnace is a growth chamber **108** in which is disposed a crucible **110**.

[0013] Noble metals and suitable alloys of noble metals may be used to manufacture the crucible. The crucible is typically manufactured from a noble metal such as ruthenium (Ru), rhodium (Rh), palladium (Pd), silver (Ag), osmium (Os), iridium (Ir), platinum (Pt), gold (Ag), or a combination thereof. Iridium is commonly used in crucibles that are used to manufacture scintillator single crystals or other high temperature materials that are not oxides or are not used for scintillation.

[0014] Disposed upon the base surface **104** is a base frame **105** upon which is located the growth chamber **108**. A conduit **135** that functions as an inlet for a first growth gas stream is disposed in the base surface **104** and the base frame **105**. The growth atmosphere (formed by the first growth gas stream) is crystal composition and crucible composition dependent. The growth atmosphere may be reducing when using hydrogen gas, slightly reducing when using nitrogen gas, oxidizing when using air, CO.sub.2, nitrogen mixed with air or oxygen, or any noble gas mixed with air or O.sub.2. The growth chamber **108** protrudes through an opening in the furnace cover **106**. The furnace cover **106** may contain internal cooling coils (not shown) through which a cooling fluid is discharged. The crucible **100** contains a melt **112** that is obtained from melting raw materials. A pull rod **114** having a seed crystal **116** disposed at its lower end is dipped into the melt **112** and then slowly moved away from the melt (moved vertically) while undergoing rotary motion. The rotary motion either clockwise or counterclockwise is used to control the interface shape of the boule while it is in the melt. The translation motion is used to control the pull rate of the boule by extracting the boule from the melt under a controlled translation rate. The translational motion refers to the linear movement of the pull rod (or boule) either upward or downward, which controls the rate at which the boule is extracted from the melt. This means that the pull rod moves vertically in a straight line to pull the crystal boule **118** out of the melt at a controlled speed.

[0015] A crystal boule is a single-crystal ingot produced by using a seed crystal to create a larger crystal, or ingot. This seed crystal is dipped into the molten raw material and slowly extracted. The melt grows on the seed crystal in a crystalline fashion. As the seed is extracted, the melt solidifies and eventually a large, cylindrical crystal boule is produced.

[0016] The growth chamber **108** contains an outer tube **107**, an inner tube **109**, a growth chamber bottom plate **128** and a growth chamber outer top plate **129**. The outer tube **107** and inner tube **109** are disposed between the growth chamber bottom plate **128** and the growth chamber outer top plate **129**. The growth chamber outer top plate **129** may be disposed on a growth chamber inner top plate **132**. The growth chamber inner top plate **132** contacts the upper portion of the outer tube **107**. The outer tube **107** is typically manufactured from quartz, while the inner tube **109** is typically manufactured from zirconia. A first O-ring seal (not shown) may be disposed between the outer tube **107** and the growth chamber bottom plate **128**. A second O-ring (not shown) is disposed between the outer tube **107** and the growth chamber inner top plate **132.** Disposed between the growth chamber bottom plate **128** and the crucible **110** is a first base plate **127** that is the subject of this disclosure. In an embodiment, the first base plate **127** contacts the bottom of the crucible **110**. The first base plate **127** replaces either all or a portion of the beads **126** that are typically located between the growth chamber bottom plate **128** and the bottom of the crucible **110**.

[0017] Disposed beneath the growth chamber bottom plate **128** and the base surface **104** of the furnace is a porous frit **130** that comprises granules or briquettes of a heat resistant material. The porous frit **130** can also permit an inert gas to pass through it.

[0018] The upper plate **129** contain two inlet ports **134** (that contact two eyepieces **120**) through which a second growth gas stream may be introduced to surround the crystal boule and the melt in the crucible **110**. The second growth gas stream may be the same as the first growth gas stream (which is described above). The eyepieces **120** may contain lenses (not shown) through which the activity in the growth chamber **108** may be viewed.

[0019] Disposed between the furnace **102** and the growth chamber **108** are induction coils **124**. The induction coils **124** (also referred to as radio-frequency (RF) coils) are used to heat the crucible and its contents and to produce the melt from which the crystal boule is manufactured. The growth chamber **108** can be moved vertically (up and down) or kept stationary with respect to the induction coils **124**.

[0020] It is to be noted in the FIG. **1** that the growth chamber bottom plate **128** and the growth chamber upper plate **129** may also contain cooling coils (not shown) through which a cooling fluid is transported.

[0021] FIG. **2** is a schematic depiction of an exemplary base plate **127** that may be used in the furnace **100** of FIG. **1**. FIG. **2** is a depiction of the base plate **127**, which as noted above comprises a plurality of alternating ridges **202**A, **202**B, **202**C, . . . **202***n* and channels **204**A, **204**B, **204**C, . . . ,**204***n* on at least one surface. The base plate **127** comprises two opposing surfaces—a first surface **302** and a second surface **304**, portions of which are parallel to each other. The outer perimeter **306** of the base plate **127** may have any geometry. It may be circular, square, rectangular, triangular, elliptical, or may include a combination of such geometries. In an embodiment, the outer perimeter **306** of the first base plate has the same geometry as the outer surface of the crucible. In a preferred embodiment, the outer perimeter **306** of the base plate **127** is circular.

[0022] The radius of the base plate may be 50 to 150% of a radius of the crucible. In a preferred embodiment, the radius of the base plate **127** (having a circular outer perimeter **306**) may vary between 5 to 75 centimeters, preferably 10 to 70 centimeters, and more preferably 15 to 60 centimeters.

[0023] At least one of these surfaces is textured to contain the plurality of alternating ridges **202**A, **202**B, **202**C, . . . , **202***n* and channels **204**A, **204**B, **204**C, . . . , **204***n*. In an embodiment, the ridges and channels extend across at least one surface of the base plate **127**. In an embodiment, the ridges and channels extend across both opposing surfaces **302** and **304** of the base plate **127**. Ridges and channels on the surface **304** (that does not contact the crucible) are optional. The ridges transfer heat to the crucible while reducing (preferably minimizing) the amount of contact between the crucible **110** and the base plate **127**. The base plate **127** has a thickness “h” of 1 centimeter to about 10 centimeters, preferably 1.5 centimeters to 5 centimeters. The channels protrude into the thickness of the base plate **127** from the surface **302** or optionally from the surface **304**. The channels may facilitate the movement of inert gases (that are introduced into the furnace) across the base plate **127**. Inert gases are circulated in the furnace to prevent oxidation of the crystal boule.

[0024] The longitudinal edges of the ridges are parallel to each other and extend across an entire surface **302** of the base plate **127**. Each ridge **202**A through **202***n* (except for an outermost ridge) has two channels **204**—one on each side of the respective ridge and each channel **204**A, **204**B, **204**C, **204**D, . . . , **204***n* (except for an outermost channel) has two ridges-one on each side of the respective channel. The upper surface **303** of the ridges **202**A, **202**B, **202**C, . . . , **202***n* are generally parallel to the bottom surface **306** of the base plate **127**.

[0025] The width of the ridges is designed to minimize the surface area contact of the base plate with the crucible while providing sufficient mechanical integrity to support the weight of the crucible and its melt/solidified contents. In an embodiment, each ridge **202**A, **202**B, **202**C, . . . , **202***n* has an upper surface having a width w.sub.1 of **0**.2 to 40 millimeters, preferably 0.4 to 20 millimeters. The width w.sub.2 of each channel **204**A, **204**B, **204**C, **204**D, . . . , **204***n* is greater than the width w of each ridge **202**A, **202**B, **202**C,., **202***n*. In another embodiment, the width of the each of the channels is less than or equal to that of each of the ridges. The channels **204**A, **204**B, **204**C, **204**D, . . . , **204***n* formed in the upper surface of the base plate **127** preferably have an outer perimeter that may be semi-circular, rectangular or square-wave like, saw-tooth like, or a combination thereof. The inner perimeter of the channels may have no internal angle or may have an interior angle that varies from 1 degree to 189 degrees. The interior angle of the inner perimeter may be 30 to 120 degrees, 40 to 110 degrees, 60 to 90 degrees, and so on.

[0026] In a preferred embodiment, each of the channels **204**A, **204**B, **204**C, **204**D, . . . , **204***n* of the base plate have a perimeter that is semi-circular of a radius of 0.5 to 60 millimeters, preferably 1 to 30 millimeters. It is to be noted that each of the channels may have an outer perimeter or shape that is different from each other. In an embodiment, some of the channels may have an outer perimeter that has a different shape from some of the other channels.

[0027] The base plate **127** is generally manufactured from a ceramic and has a melting point that is greater than the melting point of the crystalline boule. The base plate **127** may be manufactured from a metal oxide. Suitable metal oxides are silica, alumina, magnesium oxide, calcium oxide, calcium silicates, hafnium oxide, zirconia, MCrAlY (where M is either iron, nickel or cobalt, Cris chromium, Al is aluminum and Y is yttrium), or a combination thereof. In a preferred embodiment, the base plate **127** comprises zirconia, alumina, or a combination thereof.

[0028] In an embodiment, the base plate **127** is never composed of loose free-flowing particles or beads. The first base plate may either be in the form of a solid plate (a plate with minimal porosity of about 1 to 5 volume percent) or may be in the form of a porous solid (with a porosity of up to 80 volume percent, where the particulates that make up the plate are fused together and are not free flowing). In an embodiment, the base plate **127** is in the form of a single monolithic solid piece (i.e., it is in the form of a single non-divisible solid that if divided would be damaged). The base plate **127** may be monolithic and yet have a porosity in an amount of 1 to 80 volume percent, 2 to 70 volume percent, 3 to 60 volume percent, based on a total volume of the base plate. In an embodiment, the base plate 127 may have a porosity of 60 to 80 volume percent, based on a total volume of the base plate. In other words, the base plate **127** can contain a plurality of particles that are fused together to form a single piece, thus giving the base plate **127** its porosity. Inert gases may be circulated in the furnace through the porous base plate.

[0029] In another embodiment, the base plate **127** may comprise a plurality of separate monolithic parts that can be physically co-located together to form the respective base plate. In an embodiment, 2 to 6 separate monolithic parts (not shown) may be physically co-located together to form the base plate **127**. Each monolithic part can be co-located with a neighboring part using a dove tail joint, a butt joint, a mitered joint, a half-lap joint, a tongue and groove joint, a mortise and tenon joint, or a combination thereof. The FIG. **3** depicts a 4 piece base plate **127** having 4 sections **402**, **404**, **406** and **408** each of which is separately manufactured with a tongue and groove joint or half-lap joint indicated by lines **602**, **604**, **606** and **608**.

[0030] In an embodiment, the base plate is manufactured by a variety of different methods. In an embodiment, the base plate may be manufactured by mixing a slurry of the desired components of the refractory material in the proper ratios then pouring into molds followed by drying and calcination. The calcined product is then sintered to achieve the final desired product. Uniaxial or isostatic pressing may also be used at some point during the process. The slurry may comprise aluminum oxide, zirconium oxide, or a powder that comprises MCrAlY (where M is either iron, nickel or cobalt, Cris chromium, Al is aluminum and Y is yttrium), and the like.

[0031] As noted above, a solid base plate with ridges and channels such as those detailed above may be used to support the crucible during the manufacturing of a crystalline boule, while at the same time minimizing the loss of noble metals, especially iridium, which is frequently used as the crucible material.

[0032] While the invention has been described with reference to some embodiments, it will be understood by those skilled in the art that various changes may be made and equivalents may be substituted for elements thereof without departing from the scope of the invention. In addition, many modifications may be made to adapt a particular situation or material to the teachings of the invention without departing from essential scope thereof. Therefore, it is intended that the invention not be limited to the particular embodiments disclosed as the best mode contemplated for carrying out this invention, but that the invention will include all embodiments falling within the scope of the appended claims.

Claims

1. A device for manufacturing a single crystal comprising: a furnace that includes a furnace wall; a crucible disposed on a first base plate within the furnace; where the first base plate has a porosity of 1 to 80 volume percent, based on a total volume of the first base plate; and where the first base plate does not comprise free flowing particles; an induction coil disposed inside the furnace wall and outside the crucible; and a refractory lining being disposed in an annulus between the furnace wall and the crucible.

2. The device of claim 1, where the first base plate is a monolith.

3. The device of claim 1, where a surface of the first base plate contacts a bottom surface of the crucible.

4. The device of claim 3, where the surface of the first base plate that contacts the bottom surface of the crucible comprises ridges and channels.

5. The device of claim 4, where the width of the channels is greater than that of the ridges.

6. The device of claim 4, where the width of the channels is less than or equal to that of the ridges.

7. The device of claim 4, where the channels facilitate transport of inert gases in the furnace.

8. The device of claim 4, where the ridges and channels extend across an entire surface of the base plate and where the ridges and the channels are parallel to each other.

9. The device of claim 1, where the first base plate comprises alumina, zirconia, MCrAlY, or a combination thereof; where M is either iron, nickel or cobalt, Cr is chromium, Al is aluminum and Y is yttrium.

10. The device of claim 1, where the first base plate has a porosity of 1 to 5 volume percent, based on a total volume of the first base plate.

11. The device of claim 1, where the first base plate has a porosity of 60 to 80 volume percent, based on a total volume of the first base plate.

12. The device of claim 1, further comprising an outer tube that lies within the furnace such that the

induction coil is located in an annulus between the furnace wall and the outer tube.
13. The device of claim 4, where the first base plate has a radius of 50 to 150% of a radius of the crucible.
14. The device of claim 1, where an outer perimeter of the first base plate has a same geometry as an outer surface of the crucible.
15. A method of growing a high temperature material, comprising: disposing a melt within a crucible in a furnace; where the furnace includes: a furnace wall; an induction coil disposed in an annulus between the crucible and the furnace wall; where the crucible is disposed on a first base plate within the furnace; where the first base plate has a porosity of 1 to 80 volume percent, based on a total volume of the first base plate; and where the first base plate does not comprise free flowing particles; and drawing a boule from the melt within the crucible to grow the high temperature material.
16. The method of claim 15, where the first base plate is a monolith.
17. The method of claim 15, further comprising contacting a bottom surface of the crucible to a surface of the first base plate and where the surface of the first base plate that contacts the bottom surface of the crucible comprises ridges and channels.
18. The method of claim 17, further comprising discharging an inert gas into the furnace, where the inert gas is transported across the base plate via the channels.
19. The method of claim 15, further comprising discharging an inert gas into the furnace via pores in the base plate.
20. The method of claim 15, wherein the high temperature material comprises lutetium oxy-orthosilicates, lutetium yttrium oxy-orthosilicates, gadolinium oxy-orthosilicates, gadolinium aluminum gallium garnets, gadolinium-gallium-aluminum garnet, gadolinium-yttrium-gallium-aluminum garnet, gadolinium-lutetium-gallium-aluminum garnet, gadolinium-scandium-gallium garnet, gadolinium-yttrium-aluminum garnet, gadolinium-scandium-aluminum garnet, gadolinium-gallium garnet or gadolinium-yttrium-scandium-aluminum garnet.