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(54) **METHOD FOR GROWING LOW DEFECT DENSITY SILICON CARBIDE**

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(51) **Int. Cl.**⁷ **C30B 29/36**; C30B 23/00

(52) **U.S. Cl.** **117/108**; 117/89; 117/93; 117/101; 117/109; 117/902; 117/951; 117/105

(58) **Field of Search** 117/108, 109, 117/951, 105, 101, 89, 93, 902

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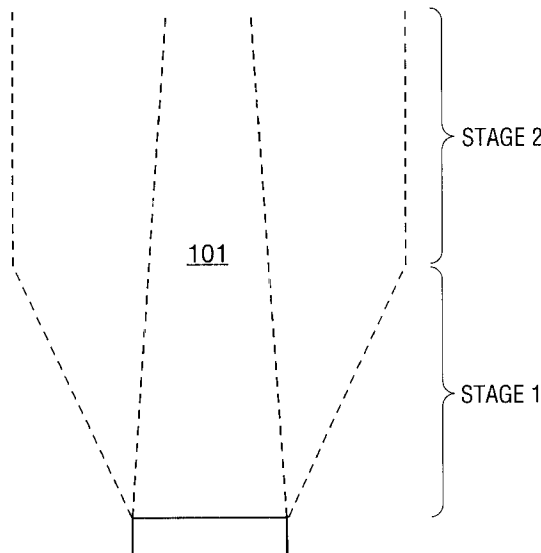
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(57) **ABSTRACT**

A low defect (e.g., dislocation and micropipe) density silicon carbide (SiC) is provided as well as an apparatus and method for growing the same. The SiC crystal, growing using sublimation techniques, is preferably divided into two stages of growth. During the first stage of growth, the crystal grows in a normal direction while simultaneously expanding laterally. Although dislocation and other material defects may propagate within the axially grown material, defect propagation and generation in the laterally grown material are substantially reduced, if not altogether eliminated. After the crystal has expanded to the desired diameter, the second stage of growth begins in which lateral growth is suppressed and normal growth is enhanced. A substantially reduced defect density is maintained within the axially grown material that is based on the laterally grown first stage material.

32 Claims, 8 Drawing Sheets



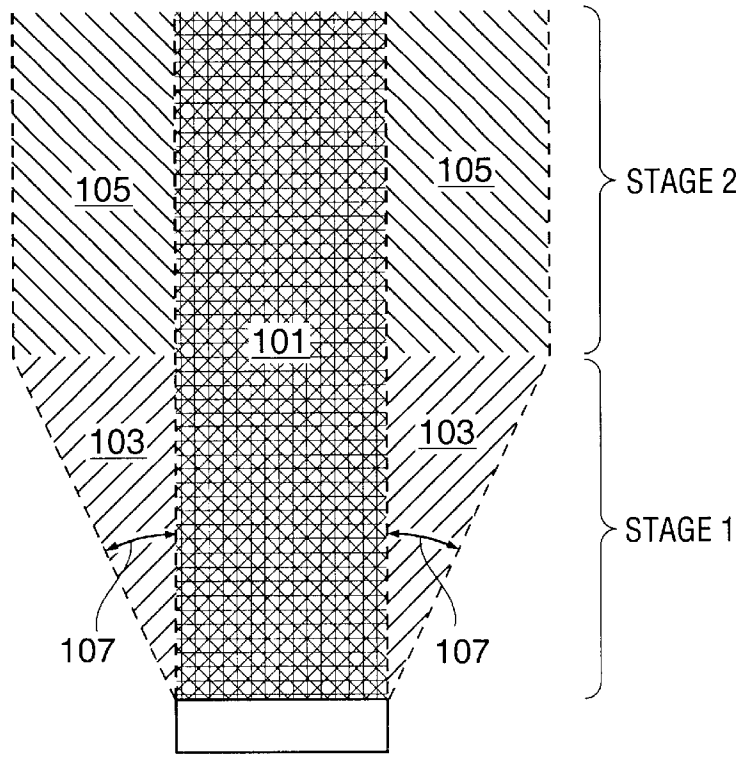


FIG. 1

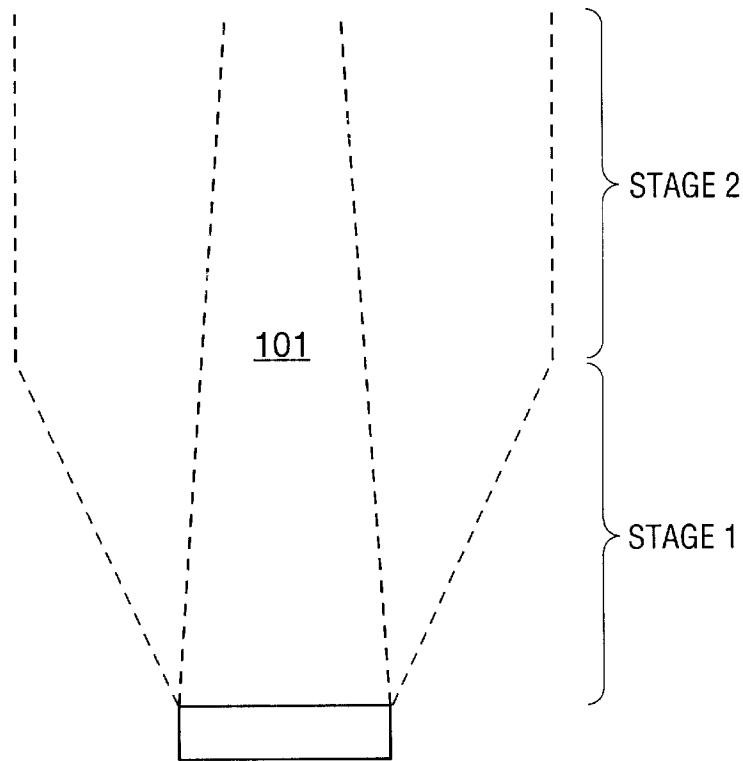


FIG. 2

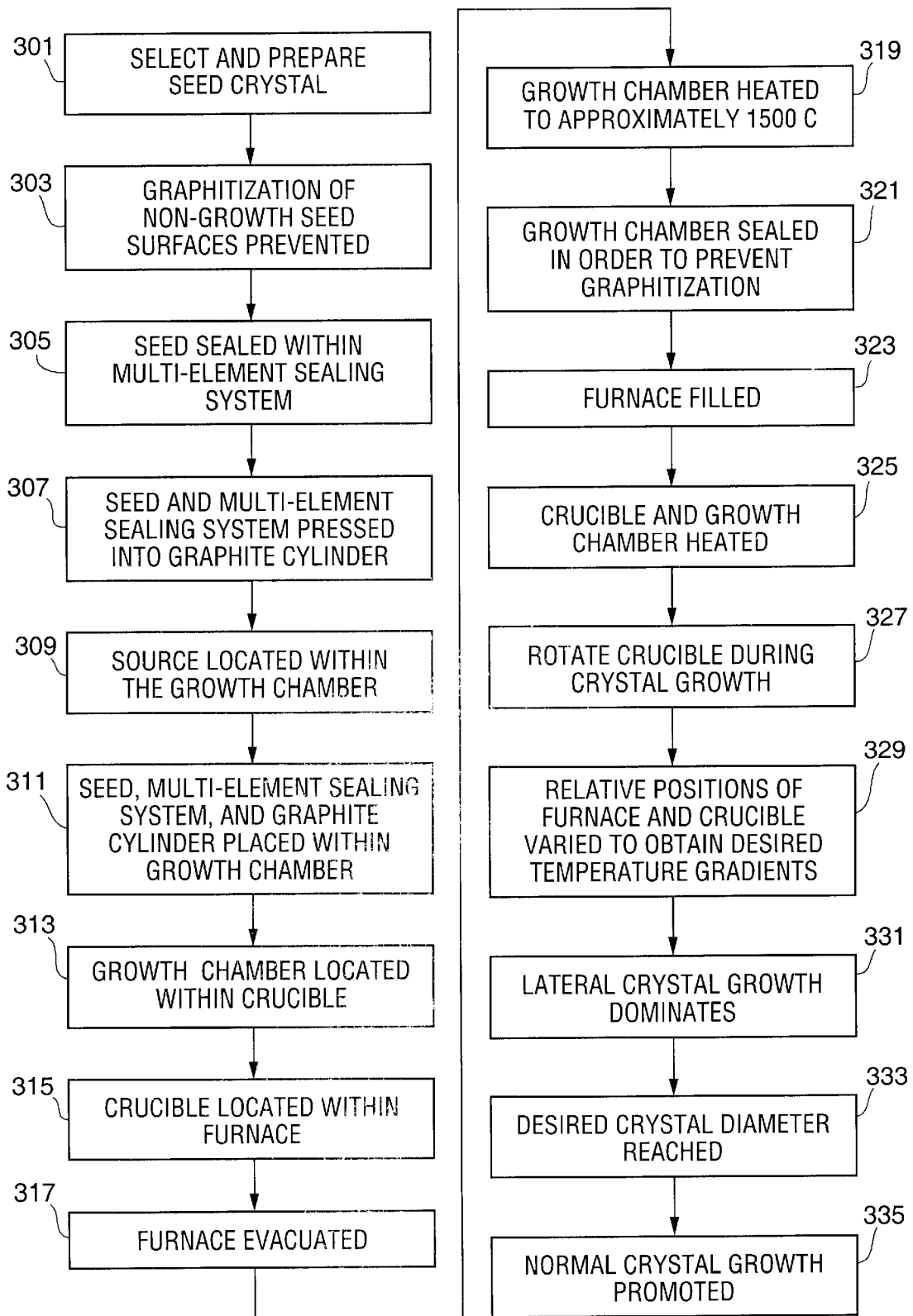


FIG. 3

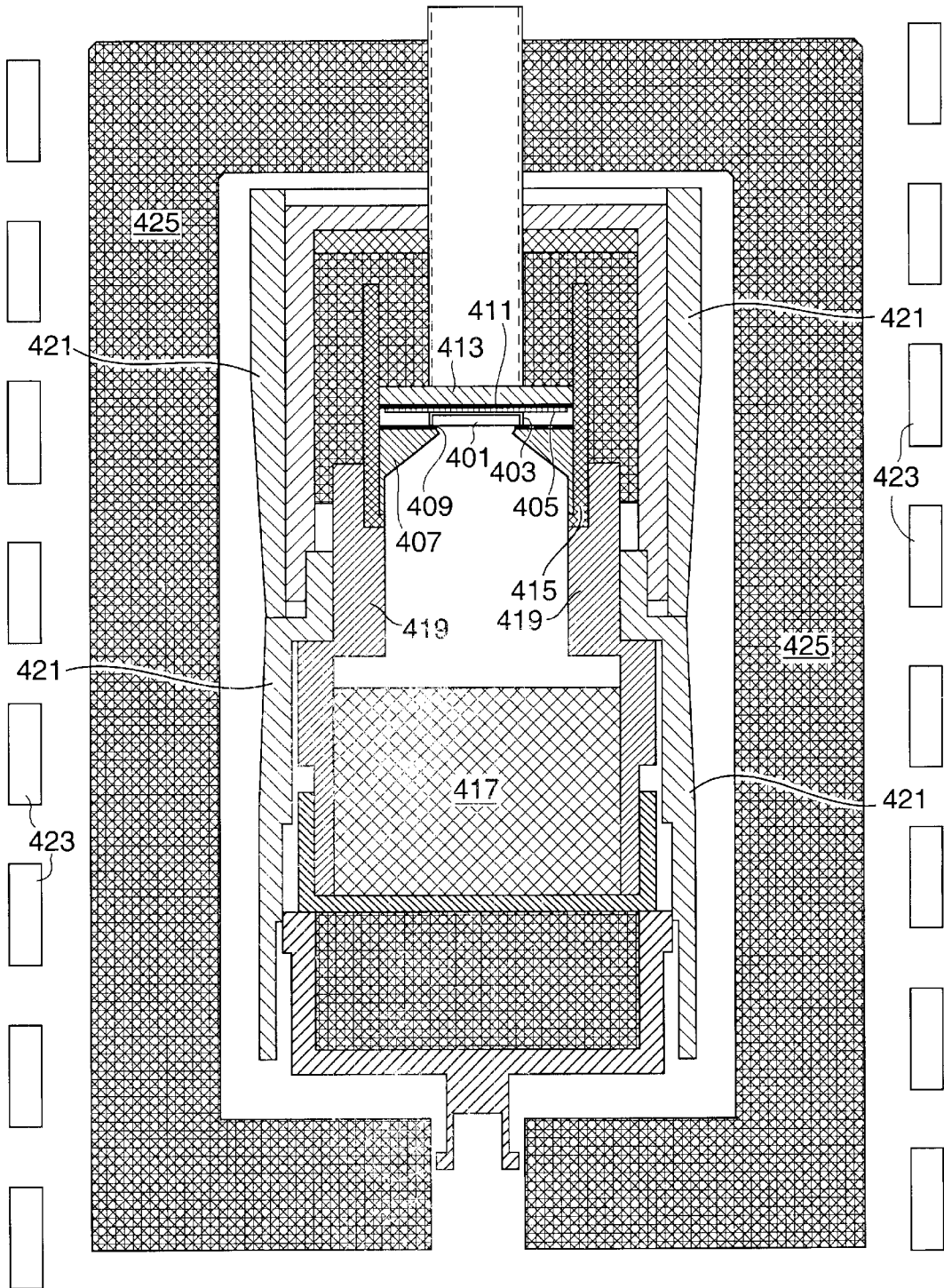


FIG. 4

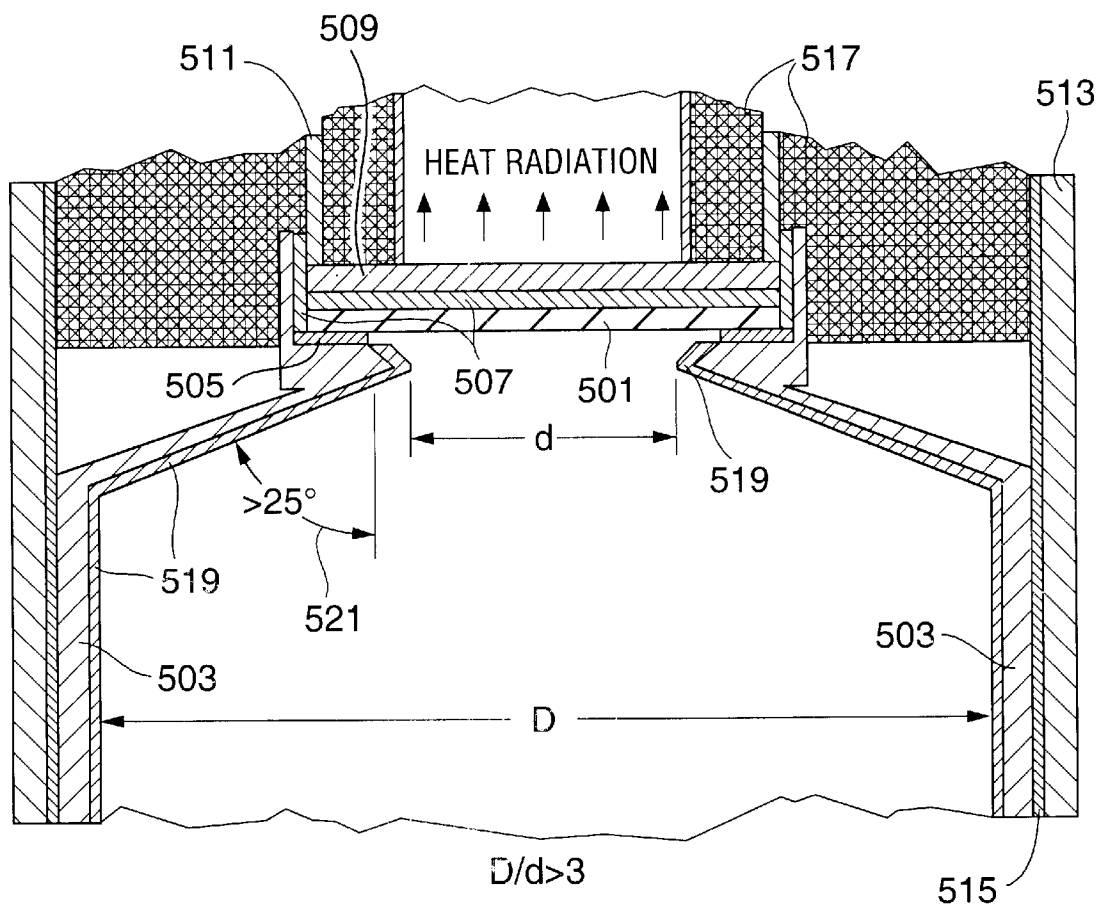


FIG. 5

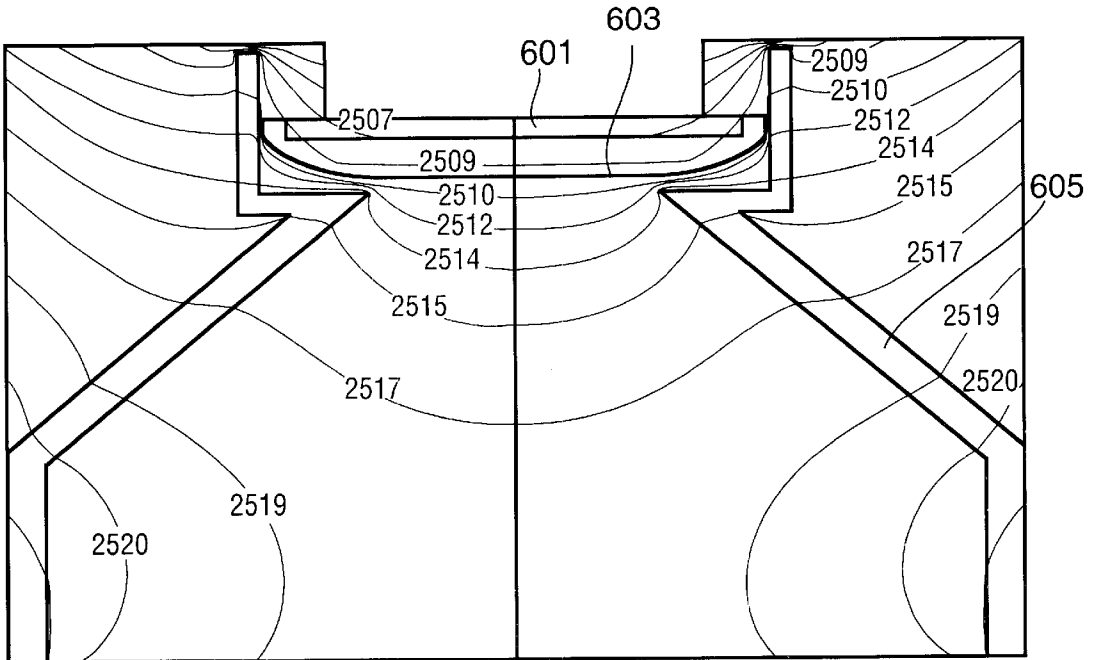


FIG. 6

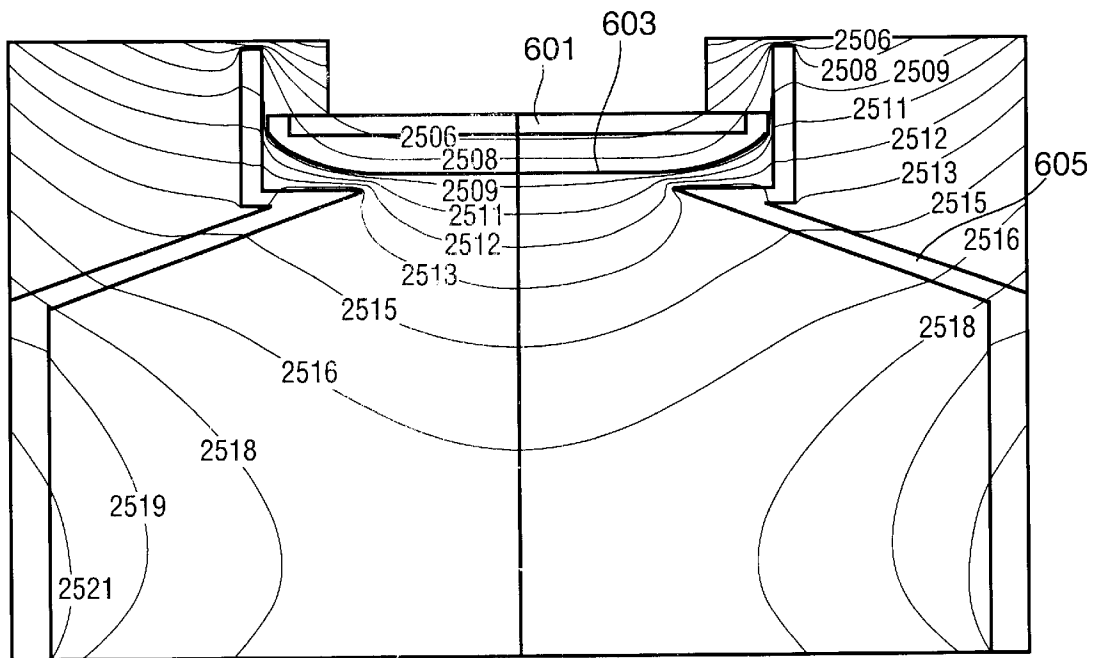


FIG. 7

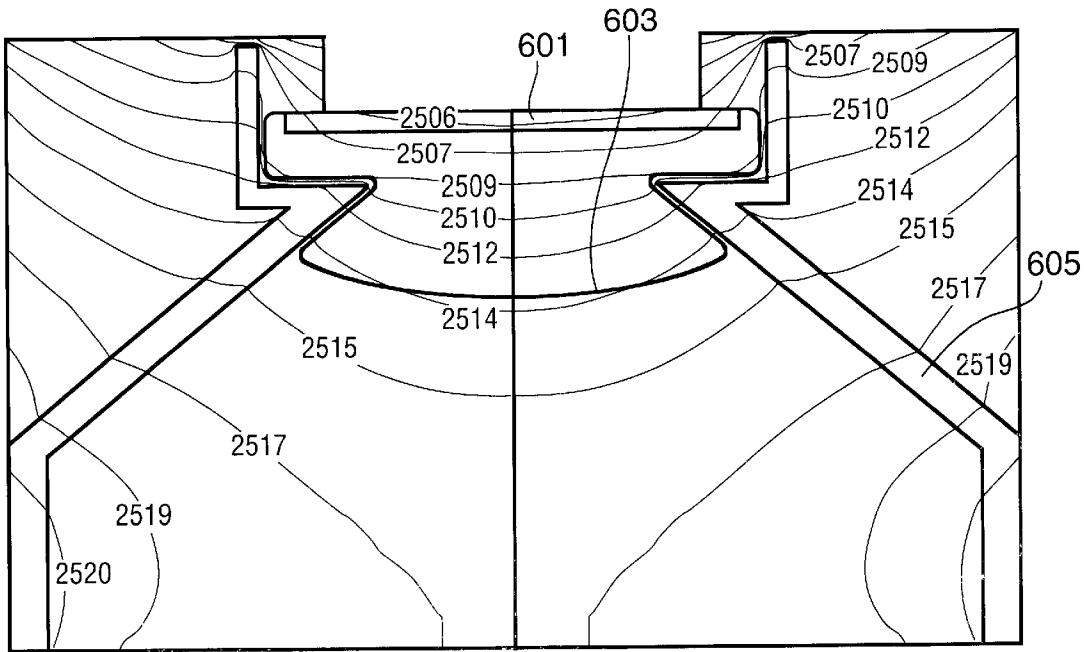


FIG. 8

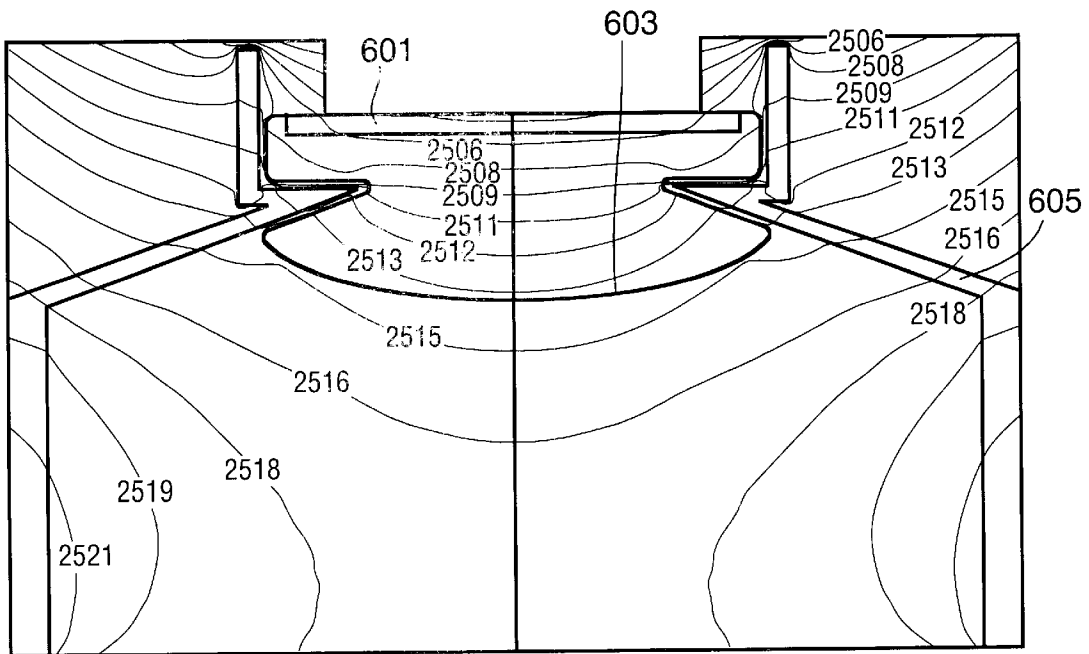


FIG. 9

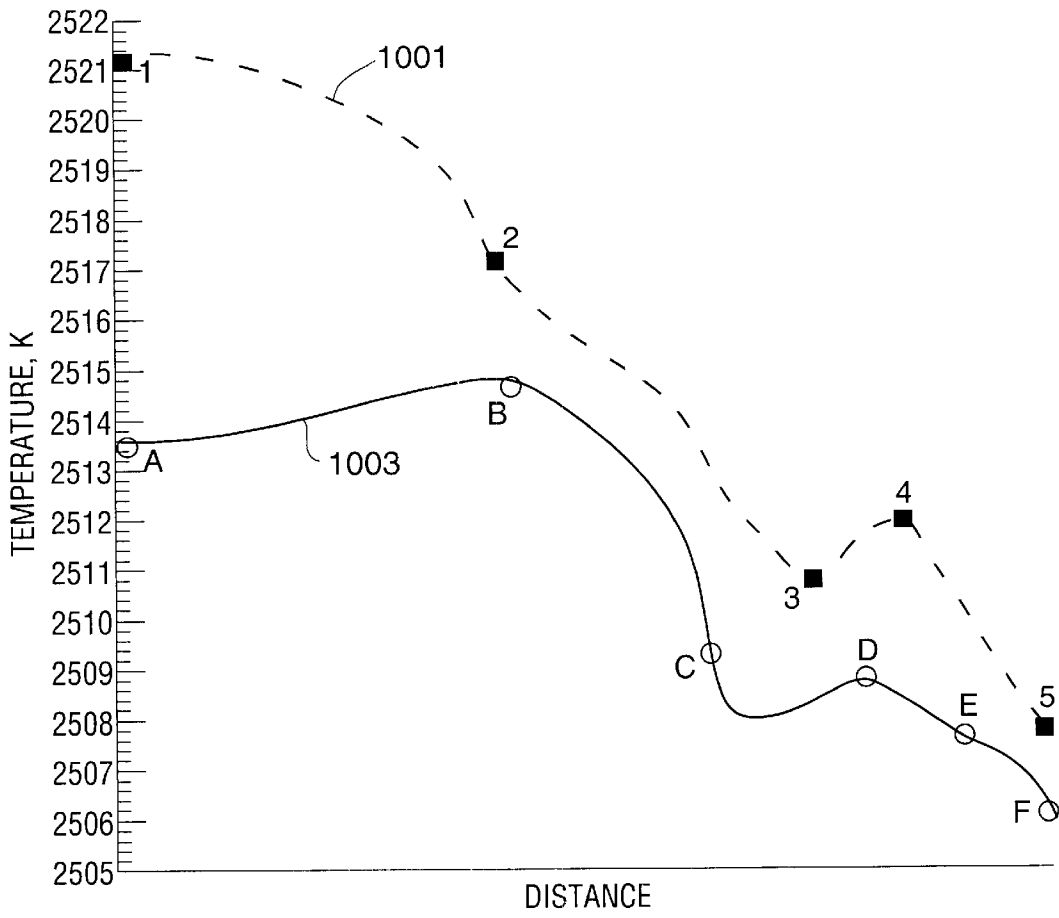


FIG. 10

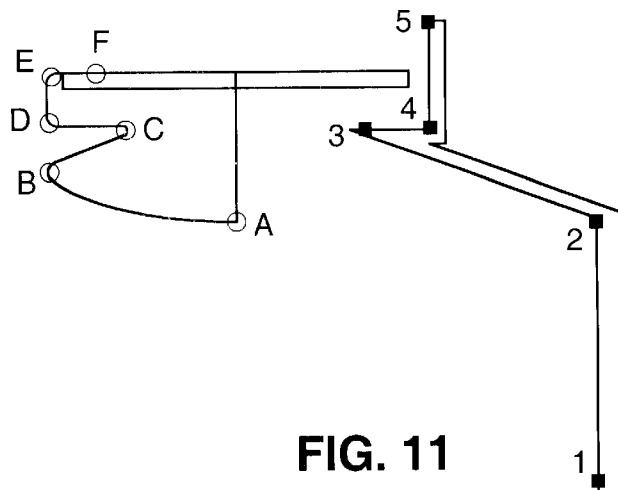


FIG. 11

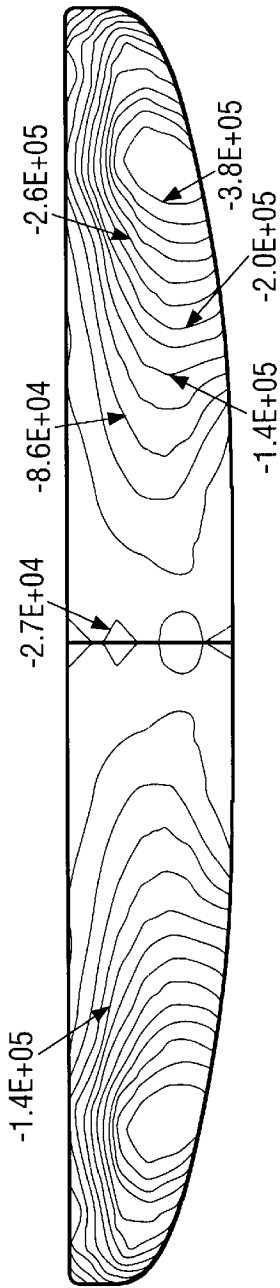


FIG. 12

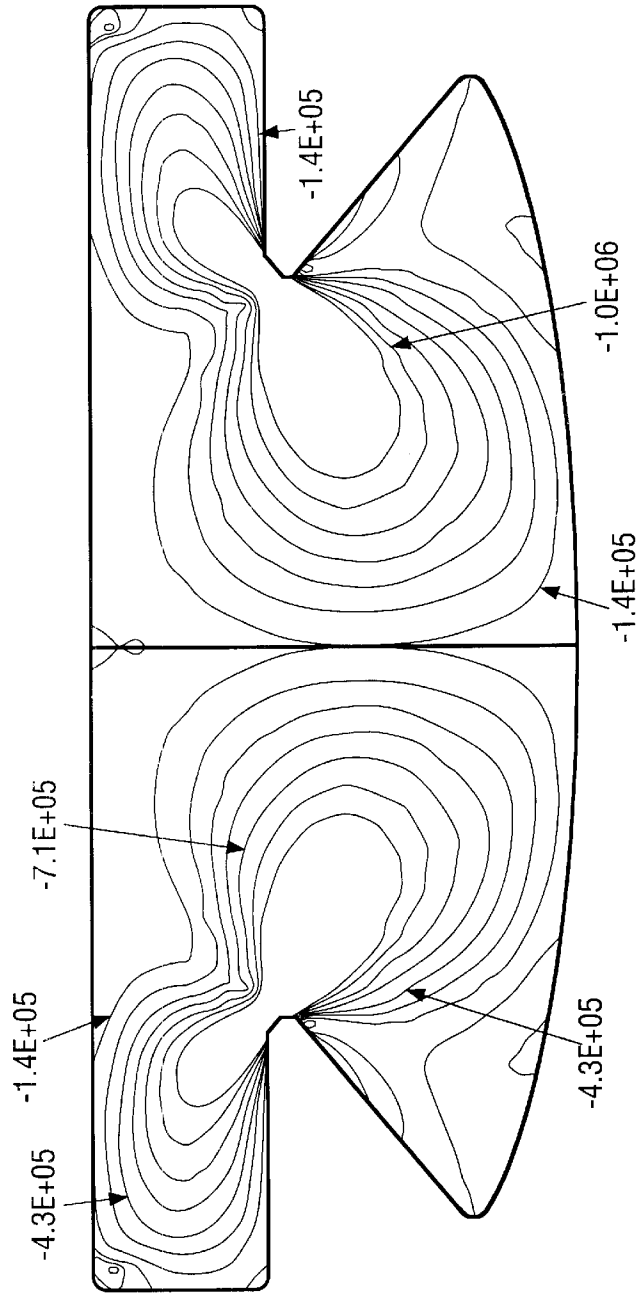


FIG. 13

METHOD FOR GROWING LOW DEFECT DENSITY SILICON CARBIDE

CROSS-REFERENCES TO RELATED APPLICATIONS

This application claims priority of U.S. Provisional Patent Application Ser. No. 60/182,553, filed Feb. 15, 2000.

FIELD OF THE INVENTION

The present invention relates generally to silicon carbide and, more particularly, to a method and apparatus for growing low defect density silicon carbide.

BACKGROUND OF THE INVENTION

Silicon carbide (SiC) has a number of characteristics that make it an ideal candidate for a variety of semiconductor applications, primarily those requiring high power handling capabilities. Arguably the most important characteristic of SiC is its indirect bandgap, resulting in relatively high recombination lifetimes and the ability to produce higher voltage junctions than those that can be produced from a direct bandgap material. The large bandgap of this material also provides for negligible current leakage up to 500° C., thereby allowing for high temperature operation without excessive leakage current or thermal runaway. The switching frequency of SiC devices is much higher than that of a device fabricated from silicon or gallium arsenide due to SiC's high breakdown strength and the resultant reduction in minority carrier storage and associated switching losses. Lastly, due to the high junction temperature and the high thermal conductivity of SiC, devices fabricated from SiC have reduced cooling requirements.

Although semiconductor devices based on SiC offer vast improvements over devices fabricated from silicon, in order to realize these improvements materials must be fabricated with much lower defect densities than have been obtainable heretofore. As noted by the authors in the 1999 article entitled *SiC Power Devices*, Naval Research Reviews, Vol. 51, No. 1 (1999), in order to scale up devices fabricated from SiC, the density of dislocations as well as the density of micropipes must be reduced. Conventional SiC material has a dislocation density between 10^5 and 10^6 per square centimeter and a micropipe density between 10^2 and 10^3 per square centimeter. Some extremely high quality SiC material has been grown with dislocation densities on the order of 10^4 per square centimeter. Unfortunately, even this dislocation density is at least an order of magnitude too high for many semiconductor applications. *Id.* at page 21.

U.S. Pat. No. 5,679,153 discloses a technique of growing SiC epitaxial layers using liquid phase epitaxy in which the density of micropipes is substantially reduced or eliminated. In one aspect of the disclosed technique, an epitaxial layer of SiC is formed on a bulk single crystal of SiC, the epitaxial layer being of sufficient thickness to close micropipe defects propagated from the bulk crystal. In order to form an electronically active region for device formation, a second epitaxial layer is formed on the first epitaxial layer by chemical vapor deposition. Based on this technique, SiC layers having micropipe densities of between 0 and 50 micropipes per square centimeter on the surface were claimed.

Although techniques have been disclosed to achieve SiC materials with low micropipe densities, these techniques do not lend themselves to growing bulk materials, i.e., materials that are at least a millimeter thick or more preferably, at least

a centimeter thick. Additionally, these techniques do not impact the dislocation densities of the material. Accordingly, what is needed in the art is a technique of growing bulk SiC material with defect densities on the order of 10^3 per square centimeter, more preferably on the order of 10^2 per square centimeter, and even more preferably on the order of 10 or less dislocations per square centimeter. The present invention provides such a technique and the resultant material.

SUMMARY OF THE INVENTION

In accordance with the invention, a low defect density silicon carbide (SiC) is provided as well as an apparatus and method for growing the same. The SiC crystal, grown using sublimation techniques, is divided into two stages of growth. During the first stage of growth, the crystal grows in a normal direction while simultaneously expanding laterally. Preferably during this stage the ratio of the lateral growth rate to the axial growth rate is between 0.35 and 1.75. Although dislocations and other material defects may propagate with in the axially grown material, defect propagation and generation in the laterally grown material are substantially reduced, if not altogether eliminated. After the crystal has expanded to the desired diameter, the second stage of growth begins in which lateral growth is suppressed and normal growth is enhanced. Preferably during this stage the ratio of the lateral growth rate to the axial growth rate is between 0.01 and 0.3, and more preferably between 0.1 and 0.3. A substantially reduced defect density is maintained within the axially grown material that is based on the laterally grown first stage material. Preferably during this stage the ratio of the lateral growth rate to the axial growth rate is between 0.01 and 0.3, and more preferably between 0.1 and 0.3. A substantially reduced defect density is maintained within the axially grown material that is based on the laterally grown first stage material.

In one aspect of the invention, a SiC material is provided with a low defect density, defects including both dislocations and micropipes. The defect density in the grown SiC is less than 10^4 per square centimeter, preferably less than 10^3 per square centimeter, more preferably less than 10^2 per square centimeter, and still more preferably less than 10 per square centimeter. In at least one embodiment, SiC is grown comprised of an axially grown region and a laterally grown region, the laterally grown region having the desired low defect density. In another embodiment of the invention, the SiC is comprised of a central region having a first defect density and a perimeter region encircling the central region that has a second defect density. The second defect density is substantially less than the first defect density and is less than 10^3 per square centimeter, preferably less than 10^2 per square centimeter, and more preferably less than 10 per square centimeter. In another embodiment of the invention, the SiC material is comprised of a SiC seed crystal, a first crystalline growth region initiating at a growth surface of the SiC seed crystal and following an axial growth path, and a second crystalline growth region of the desired defect density initiating at a growth surface of the SiC seed crystal and following a laterally expanding growth path. The laterally expanding growth path is at an angle of at least 25 degrees, and preferably at least 45 degrees, from the normal, i.e., axial, growth path.

In another aspect of the invention, a method of growing a SiC material with a low dislocation density is provided. In at least one embodiment, a SiC seed crystal is introduced into a sublimation system wherein both axial and lateral crystal growth is promoted, at least during one stage of growth. Propagation of dislocation defects, including

micropipes, from the seed crystal into the laterally grown crystal is substantially reduced as is generation of dislocation defects within this region. In at least another embodiment of the invention, a SiC seed crystal is introduced into a sublimation system and heated to a temperature sufficient to cause sublimation. Temperature gradients within the sublimation system as well as temperature differential between the crystallization growth front and adjacent surfaces promote a first stage of free space crystal expansion wherein the crystallization front expands both axially and laterally followed by a second stage of free space crystal expansion wherein the crystallization front expands axially while lateral expansion is suppressed.

In another aspect of the invention, an apparatus for use in growing a SiC material with a low dislocation density is provided. In at least one embodiment of the invention, the apparatus includes a ring element that promotes lateral crystal expansion, preferably through the use of a conical surface. The ring element may also be used to shield the edge of the SiC seed from the growth process. The ring element may also include a second surface, preferably conical, that promotes lateral crystal contraction. Preferably the ring element inner surfaces are comprised of either Ta_xC_y or Nb_xC_y . In at least one embodiment of the invention, the apparatus also includes a graphite heat sink coupled to a non-growth surface of the SiC seed crystal, a growth chamber with inner surfaces preferably comprised of either Ta_xC_y or Nb_xC_y , and means for applying temperature gradients to the crucible.

A further understanding of the nature and advantages of the present invention may be realized by reference to the remaining portions of the specification and the drawings.

BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 schematically illustrates the regions of defect free crystal growth in accordance with the invention;

FIG. 2 schematically illustrates a reduction in the core region during crystal growth;

FIG. 3 illustrates the basic methodology applied to achieve low defect density SiC;

FIG. 4 illustrates the preferred design of the sublimation system;

FIG. 5 illustrates a detailed cross-section of the critical growth region of the preferred design of the invention;

FIG. 6 illustrates the calculated temperature distribution within the growth cell and in the growing crystal for a ring element cone angle of 45 degrees and a growth period of approximately 4 hours;

FIG. 7 illustrates the calculated temperature distribution within the growth cell and in the growing crystal for a ring element cone angle of 70 degrees and a growth period of approximately 4 hours;

FIG. 8 illustrates the calculated temperature distribution within the growth cell and in the growing crystal for a ring element cone angle of 45 degrees and a growth period of approximately 22 hours;

FIG. 9 illustrates the calculated temperature distribution within the growth cell and in the growing crystal for a ring element cone angle of 70 degrees and a growth period of approximately 22 hours;

FIG. 10 illustrates a one dimensional temperature distribution over the ring element cell wall and the crystal interface;

FIG. 11 illustrates the ring element wall locations and the crystal interface locations that correspond to the data points shown in FIG. 10;

FIG. 12 illustrates the distribution of the principal component of the thermal elastic stress tensor as computed for the temperature distributions shown in FIG. 6; and

FIG. 13 illustrates the distribution of the principal component of the thermal elastic stress tensor as computed for the temperature distributions shown in FIG. 8.

DESCRIPTION OF THE SPECIFIC EMBODIMENTS

The dislocations in (0001) silicon carbide (SiC) seed crystals are primarily threading and screw dislocations in the <0001> crystal direction. Micropipe defects are basically screw dislocations with a Burger's vector that is so large that the core of the screw is empty. The inventors have found that by growing a crystal under the appropriate conditions in the radial direction (i.e., lateral direction) rather than the axial direction, the multiplication of <0001> dislocations is suppressed. Accordingly, under the appropriate conditions, a defect free SiC crystal can be grown using sublimation techniques.

In the preferred embodiment of the invention, as illustrated in FIG. 1, crystal growth is divided into two stages. During the first stage of growth, the crystal grows in a normal direction (i.e., vertically) while simultaneously expanding laterally. Preferably during this stage the ratio of the lateral growth rate to the axial growth rate is between 0.35 and 1.75. After the crystal has expanded to the desired diameter, lateral growth is suppressed while normal growth is enhanced (i.e., the second stage of growth). Preferably during this stage the ratio of the lateral growth rate to the axial growth rate is between 0.01 and 0.3, and more preferably between 0.1 and 0.3. In the preferred embodiment this ratio is not too low, thereby achieving a slightly convex growth surface and avoiding singularization of this surface. Lateral crystal growth is limited by the size of the crucible, which, in turn, is primarily limited by the ability to achieve the required temperature gradients.

According to the invention, although dislocations and other material defects propagate within core region **101**, defect propagation and generation is substantially reduced, if not altogether eliminated, in the laterally grown area **103**. Additionally, a substantially reduced defect density is maintained within the axially grown material that is based on the laterally grown material, i.e., regions **105**. Preferably the growth conditions are chosen to reduce the size of core region **101** during growth as illustrated in FIG. 2.

In order to achieve defect free lateral crystal growth, the inventors have found that preferably a number of conditions are met. These conditions, discussed in further detail below, include:

- (i) High quality seed crystal—Preferably the growth surface of the seed crystal is defect free, thereby minimizing the propagation of defects within the core region of the grown crystal.
- (ii) Shielding the back surface of the seed crystal to prevent initiation of dislocations and other microscopic defects (e.g., planar defects) which can propagate through the crystal and impact the quality of the grown crystal.
- (iii) Selection of the proper growth angle—Generally angle **107** in FIG. 1 must be greater than 25 degrees, and preferably greater than 45 degrees. This angle is primarily determined by two factors; first, the vertical temperature gradient between the source and the seed crystal and second, the lateral temperature gradient between the center of the crucible and the crucible walls.

- (iv) Selection of an appropriate seed crystal diameter—Generally the diameter of the seed crystal should be less than 30 percent of the diameter of the crystal to be grown.
- (v) Prevention of polycrystalline growth—Requires the elimination of contact between the laterally growing material and the crucible side-walls thus insuring free-space expansion of the crystal. This is achieved by maintaining a temperature differential between the laterally growing crystal and the crucible side-walls.

FIG. 3 illustrates the basic methodology applied to achieve defect free SiC while corresponding FIG. 4 illustrates the preferred design for the furnace and crucible. Initially a SiC seed crystal **401** is selected and prepared. (Step **301**) Seed crystal **401** can be grown using any of a number of well known techniques (e.g., Lely method). In the preferred embodiment a seed crystal with minimal defects is used, typically on the order of 10^5 per square centimeter or less. Also preferably the seed crystal has minimal, if any, micropipe defects. The ratio of the diameter of seed crystal **401** to the diameter of the crystal to be grown is preferably less than 0.3.

In the preferred embodiment of the invention, surface mechanical defects are removed from the surface of the seed crystal using conventional surface preparation techniques, e.g., grinding, polishing, and chemical etching. In this embodiment, approximately 50 microns is removed although the removal of additional material in excess of a 50 micron layer may be required in order to achieve the desired surface. Preferably the surface finish has an RMS roughness of 50 Angstroms or less.

During crystal growth, it is important to prevent graphitization. (Step **303**) Accordingly, the back surface of seed crystal **401** is preferably shielded during evaporation. The preferred method of shielding the non-growth surfaces of seed crystal **401** is to place the growth surface of the crystal onto the flat, polished surface of a tantalum disk. Then the wafer is annealed in vacuum for a few minutes at 1700 to 1750° C., yielding a dense graphite layer **403** on those seed crystal surfaces not in contact with the tantalum disk. Once formed, back surface graphite layer **403** is coupled to a holder **405**. Preferably holder **405** is comprised of graphite or pyrographite and a graphite based glue is used to couple seed crystal **401** to holder **405**, the glue eliminating voids in the joint between the two surfaces. In addition to helping to prevent graphitization, the above-described crystal seating process also prevents localized temperature non-uniformities in the seating area that arise from having voids between the back surface of the seed crystal and the crucible lid (e.g., the seed holder).

In an alternate embodiment, the dense graphite layer **403** is grown on all seed crystal surfaces and subsequently removed from the growth surface, for example using an epi-polishing technique.

In order to shield the non-growth seed surfaces of the seed crystal from evaporation, in the preferred embodiment of the invention seed crystal **401** is sealed within a multi-element system that includes a ring element **407**. (Step **305**) Ring element **407** is not only integral to the sealing system, it also helps to shape the desired lateral growth of the crystal. The outer surface of element **407** is cylindrical while the inner surfaces are generally conical and coated with Ta_xC_y or Nb_xC_y . Preferably the inner surfaces of element **407** are comprised of a pair of conical surfaces as shown in FIG. 4.

Seed crystal **401** is pressed and sealed to ring element **407** using a gas impermeable graphite foil **409**. As shown, the inner diameter of the portion of ring element **407** adjacent to

seed crystal **401** as well as the interposed gas impermeable foil **409** is smaller than the outer diameter of crystal **401**. Accordingly, the edge of crystal **401** is unexposed, thus controlling growth of crystal defects that typically would originate at the crystal edge, the crystal edge defined as the juncture of the crystal face surface and the crystal side surface. In addition to preventing exposure of the crystal edge, ring element **407** provides a means of achieving the desired lateral temperature gradient and thus, as previously noted, provides a means of controlling the lateral growth of the crystal.

In the preferred embodiment of the invention, the inner surface of ring element **407** is comprised of a pair of conical surfaces, the pair of conical surfaces providing a slight necking down of the crystal growth surface. As such, prior to undergoing lateral expansion, the crystal growth surface undergoes an initial period of contraction. In an alternate embodiment, ring element **407** is comprised of a single conical inner surface that defines the laterally expanding crystal surface. In an alternate embodiment, ring element **407** is comprised of a single conical inner surface that defines the laterally expanding crystal surface and a non-conical inner surface immediately adjacent to the seed crystal that insures that the crystal growth interface initially undergoes a period of contraction prior to undergoing lateral expansion. As in the preferred embodiment, in the alternate embodiments the inner diameter of the portion of element **407** in contact with foil **409** (and adjacent to crystal **401**) is smaller than the outer diameter of seed crystal **401**, thus controlling growth of edge defects.

A second gas impermeable graphite foil **411** is used to seal the back surface of seed crystal **401** and holder **405** to a graphite heat sink **413**. Foil **411** helps to achieve a good thermal contact between crystal **401**/holder **405** and heat sink **413**, the thermal contact preferably being continuous across the entire heat sink interface. As shown, the outer diameter of graphite heat sink **413** is substantially equal to the outer diameter of ring element **407**. The stack, comprised of heat sink **413**, holder **405**, seed crystal **401**, ring element **407**, and seal **409** and **411**, is press fit within a thin-wall graphite cylinder **415** (step **307**), thereby preventing reactive gases such as Si, Si_2C , and/or SiC_2 from reaching the non-growth surfaces of seed crystal **401**.

A source **417** is placed within a growth chamber **419**. (step **309**) Seed crystal **401**, the multi-element sealing system and graphite cylinder **415** are then located within the growth chamber. (Step **311**) Growth chamber **419** is fabricated from Ta_xC_y , Nb_xC_y , or graphite. If graphite is used for growth chamber **419**, the inner surfaces of the chamber are coated with either Ta_xC_y or Nb_xC_y . Preferably the distance between the seed crystal growth surface and the top of the source material **417** is less than 30 percent of the diameter of source **417**, thus allowing quasi-equilibrium vapor phase conditions to be maintained.

Source **417** is such a manner as to suppress the formation of source particles in the gas phase during crystal growth. In the preferred embodiment of the invention, this goal is achieved by annealing electronic grade SiC powder or a mixture of Si and C powders at a temperature of between about 2100 and 2500°C. for approximately 1 hour. As a result of the annealing process, a dense deposit is formed that eliminates particle formation during crystal growth. In order to obtain doped SiC crystals using the invention, the desired dopants and/or impurities (e.g., nitrogen, boron, aluminum, indium, vanadium, molybdenum, scandium, chromium, iron, magnesium, tin, and zirconium) are included in source **417**.

Growth chamber 419 is placed within a two-piece graphite crucible 421 (step 313), the shape (e.g., the tapered portions) of which is designed to provide the temperature gradients described in further detail below. In order to prevent graphitization, a requirement of the present invention, the stoichiometry of the vapor within the growth chamber, i.e., the ratio of silicon to carbon, must remain relatively constant during crystal growth. One method of realizing this objective is to minimize material losses. Accordingly, in the preferred embodiment of the invention, the rate of material loss during the growth process is maintained at a level of less than 0.5 percent of the initial source weight per hour. In particular this rate loss is achieved by first locating graphite crucible 421 within a high temperature furnace 423, preferably an RF induction furnace as shown. (Step 315) Graphite foam 425 is used to suppress heat losses from the furnace. The furnace, along with the growth chamber, is next evacuated down to a pressure of 10^{-5} torr or less (step 317) and then heated to a temperature of approximately 1500° C. (step 319). Chamber 419 is then sealed, preferably using different types of graphite with different coefficients of thermal expansion, in order to prevent graphitization. (Step 321)

After chamber sealing, the furnace is filled with pure argon or argon with traces of nitrogen. (Step 323) To obtain the desired resistivity within the grown crystal, the partial pressure of the gas filled furnace is maintained within a range of 10^{-1} to 10^{-4} torr. Crucible 421 and chamber 419 are then heated to a temperature of between 1900 and 2400° C. at a rate of between 6 and 20° C. per minute. (Step 325)

During crystal growth, crucible 421 is axially rotated at a rate of approximately 1 to 5 revolutions per minute. (Step 327) As the crystal grows, the required temperature gradients are achieved, at least in part, by altering the relative positions of crucible 421 and furnace 423. (Step 329) Typically the rate of movement is approximately equivalent to the rate of crystal growth, i.e., between 0.1 and 1.5 millimeters per hour.

Preferred Crystal Growth Methodology

In addition to the method and apparatus described above, the inventors have found that certain growth methodologies are preferred. As previously noted, free-space expansion of the crystal during growth is critical to achieving defect free SiC. Accordingly, it is important to prevent the formation of polycrystalline deposits on all surfaces that surround seed 401, such surfaces including ring element 407, seed holder 405, graphite cylinder 415, and growth chamber 419. Insuring that the temperature of the surface in question is higher than that of the seed is the preferred technique for preventing polycrystalline deposits. At the same time, however, it is important that the temperature differential between the seed and the adjacent surfaces not be too great, otherwise lateral crystal growth may be deterred. Accordingly, the inventors have found that the temperature differential between the crystallization front and the adjacent surface before the crystallization front should be in the range of 1 to 5° C.

The inventors have found that during the period of time in which the crystal is undergoing lateral expansion, a temperature drop of between 5 and 25° C., and preferably between 5 and 10° C., should be maintained between seed 401 and source 417. This temperature differential aids in the suppression of normal (i.e., non-lateral) crystal growth. Preferably there is a lateral dependence to the temperature differential such that the smallest temperature differential occurs at the center of the seed, increasing with lateral distance. As a result, a convex crystallization growth front is formed which aids in the elimination of micropipe propagation.

As previously described, preferably the angle between the normal crystal growth and the lateral crystal growth (e.g., angle 107 of FIG. 1) is greater than 25 degrees. If the angle is less than 25 degrees, the defects of seed crystal 401 and any defects that may be generated during the initial crystal growth will continue to propagate throughout the newly grown crystal. If the angle is greater than 45 degrees, as in the preferred embodiment of the invention, typically all of the defects will move towards the lateral surface and, once the lateral surface is reached, not participate further in the growth process. In those cases in which not all of the defects are eliminated from participation in the growth process, the defect density in the laterally grown material is typically on the order of 10^2 per square centimeter or less, and more typically on the order of 10 per square centimeter or less. In the intermediate situation in which the angle is between 25 and 45 degrees, the expansion of defects into the laterally growing crystal body is typically observed. If seed crystal 401 is of a high quality, however, angles within this range may yield crystals of sufficiently low defect density.

Initially, lateral crystal growth dominates (step 331), the laterally grown crystal being free of micropipes and having a defect (e.g., dislocations, micropipes) density less than 10^4 per square centimeter, preferably less than 10^3 per square centimeter, more preferably less than 10^2 per square centimeter, still more preferably less than 10 per square centimeter, and still more preferably with zero defects per square centimeter. As observed, this material is free of any graphite inclusions. Crystal growth, dominated by lateral crystal growth, continues until the desired crystal diameter is reached, this crystal diameter being defined by the growth chamber in general, and for the embodiment illustrated in FIG. 5, by ring element 407. Once the crystal reaches the desired diameter (step 333), the vertical temperature gradient is changed to promote normal, i.e., vertical, crystal growth (step 335). In order to achieve the desired change in the temperature gradient, the relative positions of furnace 423 and crucible 421 are changed. In the preferred embodiment of the invention, furnace 423 is an inductive furnace and the coils of the furnace are moved relative to crucible 421. Alternately, or in addition to changing the relative positions of the furnace and the crucible, the temperature within portions of the furnace may be changed. Preferably the axial temperature gradient, i.e., the gradient between the source and the growth surface, is in the range of 10 to 50° C. per centimeter, yielding the desired normal growth rate of between 0.4 and 1.5 millimeters per hour.

During the last stage of crystal growth it is important to prevent considerable lateral crystal expansion. It is also important, as previously described, to prevent SiC deposits from forming on the crucible side walls (e.g., the side walls of ring element 407, graphite cylinder 415, and growth chamber 419). Accordingly, a higher side wall temperature is maintained relative to the temperature of the seed crystal, preferably the temperature difference being at least 10° C., more preferably between 10 and 30° C., and still more preferably between 10 and 15° C. The higher side wall temperature radiatively heats the sides of the growing crystal, thereby achieving hotter crystal side walls than the normal growth surface of the crystal. As a result, all of the vapor species are consumed at the normal growth surface of the crystal and growth on the crucible side walls is suppressed. Additionally, this temperature difference insures that the growing crystal does not come into contact with the crucible side walls, such contact being a major source of defects.

The inventors have also found that the temperature gradient in the growing crystal must be maintained at a rela-

tively low number, preferably on the order of 5° C. per centimeter or less. If the temperature gradient becomes too large, strain is created within the growing crystal, resulting in the formation of dislocations or other defects.

Detailed Growth Region

FIG. 5 is a cross-sectional view of the critical growth region in the preferred embodiment of the invention. In this embodiment the SiC seed crystal 501 is held within a portion of ring element 503. A graphite foil ring 505 is interposed between ring element 503 and the growth surface of crystal 501, foil ring 505 sealing the seed crystal to the ring element. The side and back surfaces of crystal 501 are covered with a graphite foil 507. A graphite disk 509 is coupled to seed crystal 501 via graphite foil 507. The primary purpose of disk 509 and interposed graphite foil 507 is to aid in the removal of heat from crystal 501. Additionally, disk 509 provides a support surface for crystal 501 as well as a means for conveniently applying pressure to the crystal with graphite ring 511, thereby achieving a seal between the crystal and element 503. Graphite foils 505 and 507 are typically between 0.25 and 0.80 millimeters thick.

Ring element 503 is preferably press fit within a graphite cylinder 513. Graphite foil 515, typically between 0.25 and 0.80 millimeter thick, is preferably interposed between the outer wall of ring element 503 and the inner wall of cylinder 513, thus helping to achieve a good pressure seal. Graphite foam 517 is used to suppress heat loss from the furnace.

In this embodiment of the invention, ring element 503 is fabricated from graphite with inner surfaces 519 coated with Ta_xC_y , or Nb_xC_y . Diameter D, the largest inner diameter of element 503, is 30 millimeters although there are no major limitations to increasing this diameter, thereby yielding a larger grown crystal. Diameter d, the smallest inner diameter of element 503 is selected such that the ratio D/d is greater than 3. Angle 521 is selected, as previously disclosed, to be greater than 25 degrees and preferably less than 90 degrees.

Thermal Analysis

FIGS. 6–10 provide calculated temperature distributions within the growth cell and in the growing crystal for a specific embodiment of the invention. In FIGS. 6–9 the seed crystal is indicated as substrate 601, the crystal growth interface is indicated as surface 603, and the ring element is indicated as element 605. For purposes of this analysis, ring element 605 is comprised of a single conical surface rather than a pair of conical surfaces as shown in the ring elements of FIGS. 4 and 5.

FIGS. 6 and 7 illustrate the temperature distribution after approximately 4 hours of growth while FIGS. 8 and 9 illustrate the temperature distribution after approximately 22 hours of growth. The cone angle for the ring element in FIGS. 6 and 8 is 45 degrees while the cone angle for the ring element in FIGS. 7 and 9 is 70 degrees.

FIG. 10 illustrates a one dimensional temperature distribution over the ring element cell wall, i.e., line 1001, and the crystal interface, i.e., line 1003. The ring element wall locations and the crystal interface locations that correspond to the data points shown on lines 1001 and 1003, respectively, are shown in FIG. 11.

Thermal Elastic Stress Distribution

As disclosed above, the temperature gradient within the growing crystal must be maintained at a relatively low number, preferably 5° C. per centimeter or less. FIGS. 12 and 13 illustrate the distribution of the principal component of the thermal elastic stress tensor σ_{rz} given in Pascals, as computed for the temperature distributions shown in FIGS. 6 and 8. The illustrated thermal elastic stress component is responsible for gliding of dislocations.

The computed results indicate that the σ_{rz} value does not exceed the SiC plasticity threshold in the major portion of the crystal. Accordingly, the probability of generating dislocations within the growing crystal is negligible.

As will be understood by those familiar with the art, the present invention may be embodied in other specific forms without departing from the spirit or essential characteristics thereof. Accordingly, the disclosures and descriptions herein are intended to be illustrative, but not limiting, of the scope of the invention which is set forth in the following claims.

What is claimed is:

1. A method of growing low defect density silicon carbide, the method comprising the steps of:

5 introducing a single crystal silicon carbide seed into a sublimation system;

10 promoting axial crystal growth to form an axial growth region during a first growth stage, wherein at least a portion of a plurality of dislocation defects and micropipes originating in said single crystal silicon carbide seed propagate into said axial growth region; and

15 promoting lateral crystal growth to form a lateral growth region during said first growth stage, wherein propagation of said plurality of dislocation defects and micropipes from said single crystal silicon carbide seed into said lateral growth region is substantially reduced.

2. The method of claim 1, further comprising the step of suppressing lateral growth during a second growth stage, wherein axial crystal growth of said axial growth region and of said lateral growth region continues during said second growth stage.

3. The method of claim 1, further comprising the step of promoting lateral crystal contraction during a second growth stage, wherein said second growth stage occurs prior to said first growth stage.

4. The method of claim 1, wherein said step of promoting lateral crystal growth further comprises the step of promoting a lateral crystal growth angle of at least 25 degrees.

5. The method of claim 1, wherein said step of promoting lateral crystal growth further comprises the step of promoting a lateral crystal growth angle of at least 45 degrees.

6. The method of claim 1, wherein said plurality of dislocation defects and micropipes propagated into said lateral growth region from said single crystal silicon carbide seed or originating in said lateral growth region result in a defect density of less than 10^4 per square centimeter within said lateral growth region.

7. The method of claim 1, wherein said plurality of dislocation defects and micropipes propagated into said lateral growth region from said single crystal silicon carbide seed or originating in said lateral growth region result in a defect density of less than 10^3 per square centimeter within said lateral growth region.

8. The method of claim 1, wherein said plurality of dislocation defects and micropipes propagated into said lateral growth region from said single crystal silicon carbide seed or originating in said lateral growth region result in a defect density of less than 10^2 per square centimeter within said lateral growth region.

9. The method of claim 1, wherein said plurality of dislocation defects and micropipes propagated into said lateral growth region from said single crystal silicon carbide seed or originating in said lateral growth region result in a defect density of less than 10 per square centimeter within said lateral growth region.

10. A method of growing low defect density silicon carbide, the method comprising the steps of:

10 introducing a single crystal silicon carbide seed into a sublimation system, said single crystal silicon carbide seed comprised of a growth surface and a plurality of non-growth surfaces;

15 heating a silicon carbide source to a temperature sufficient for the silicon carbide source to sublime;

initiating a first stage of free space crystal expansion wherein a crystallization growth front laterally and axially expands to define a growing silicon carbide crystal, wherein lateral expansion is at an angle of at least 25 degrees as measured from a normal growth axis, said first stage initiating step further comprising the steps of:

maintaining a temperature differential between said growth surface and said silicon carbide source, wherein a temperature of said growth surface is between 5° C. and 25° C. lower than a temperature of said silicon carbide source;

maintaining a temperature differential in the range of 1° C. to 5° C. between said crystallization growth front and adjacent surfaces;

maintaining a temperature gradient in said growing silicon carbide crystal of 5° C. per centimeter or less; and

initiating a second stage of free space crystal expansion wherein said crystallization growth expands axially and lateral expansion is suppressed, said second stage initiating step further comprising the steps of:

maintaining an axial temperature gradient between said growth surface and said silicon carbide source in the range of 10° C. to 50° C. per centimeter;

maintaining a temperature differential between said crystallization growth front and adjacent surfaces of at least 10° C.; and

maintaining said temperature gradient in said growing silicon carbide crystal of 5° C. per centimeter or less.

11. The method of claim 10, wherein said lateral expansion is at an angle of at least 45 degrees as measured from said normal growth axis.

12. The method of claim 10, wherein said temperature of said growth surface is between 5° C. and 10° C. lower than said temperature of said silicon carbide source during said first stage initiating step.

13. The method of claim 10, further comprising the step of preventing graphitization of said plurality of non-growth surfaces of said single crystal silicon carbide seed.

14. The method of claim 13, said step of preventing graphitization of said plurality of non-growth surfaces of said single crystal silicon carbide seed further comprising the steps of:

locating said growth surface of said single crystal silicon carbide seed onto a surface of a tantalum disk; and

annealing said single crystal silicon carbide seed, wherein said step of preventing graphitization of said plurality of non-growth surfaces of said single crystal silicon carbide seed occur prior to said introducing step.

15. The method of claim 10, further comprising the step of preventing graphitization within a growth chamber of said sublimation system.

16. The method of claim 10, further comprising the step of maintaining relatively stable vapor stoichiometry during said first and second stages of free space crystal expansion.

17. The method of claim 10, further comprising the step of maintaining a material loss rate of less than 0.5 percent of an initial source weight per hour during said first and second stages of free space crystal expansion.

18. The method of claim 10, further comprising the step of removing mechanical defects from said growth surface of said single crystal silicon carbide seed prior to said step of introducing said single crystal silicon carbide seed into said sublimation system.

19. The method of claim 10, further comprising the step of axially rotating a crucible of said sublimation system at a

rate of 1 to 5 revolutions per minute during said first and second stages of free space crystal expansion.

20. The method of claim 10, further comprising the step of separating said silicon carbide source and said growth surface of said single crystal silicon carbide seed by a distance of less than 30 percent of a diameter of said source of reactive gases.

21. The method of claim 10, further comprising the step of suppressing source particle formation during said first and second stages of free space crystal expansion.

22. The method of claim 21, wherein said step of suppressing source particle formation further comprises the step of forming said source of reactive gases from annealed silicon carbide powder or an annealed mixture of silicon powder and carbon powder.

23. The method of claim 22, wherein said silicon carbide powder or said mixture of silicon powder and carbon powder is annealed at a temperature between about 2100° C. and about 2500° C.

24. The method of claim 10, further comprising the step of including a dopant or an impurity within said silicon carbide source, said dopant and said impurity selected from the group consisting of nitrogen, boron, aluminum, indium, vanadium, molybdenum, scandium, chromium, iron, magnesium, tin, and zirconium.

25. The method of claim 10, further comprising the step of coupling at least one of said plurality of non-growth surfaces of said single crystal silicon carbide seed to a heat sink.

26. The method of claim 25, wherein said step of coupling said at least one of said plurality of non-growth surfaces of said single crystal silicon carbide seed to said heat sink further comprises the step of interposing a graphite foil between said at least one of said plurality of non-growth surfaces of said single crystal silicon carbide seed and said heat sink.

27. The method of claim 10, further comprising the step of preventing exposure of an edge portion of said growth surface of said single crystal silicon carbide seed to said silicon carbide source.

28. The method of claim 27, wherein said step of preventing exposure of said edge portion of said growth surface to said silicon carbide source further comprises the step of coupling an outer region of said growth surface of said single crystal silicon carbide seed to a ring element.

29. The method of claim 28, wherein said step of coupling said outer region of said growth surface of said single crystal silicon carbide seed to said ring element further comprises the step of interposing a graphite foil between said outer region of said growth surface of said single crystal silicon carbide seed and said ring element.

30. The method of claim 10, further comprising the step of preventing exposure of said plurality of non-growth surfaces of said single crystal silicon carbide seed to said silicon carbide source.

31. The method of claim 10, further comprising the step of sealing a growth chamber of said sublimation system prior to initiating said first and second stages of free space crystal expansion.

32. The method of claim 31, said step of sealing said growth chamber further comprising the steps of:

evacuating said growth chamber to a pressure of 10⁻⁵ torr or less;

heating said growth chamber to a temperature of approximately 1500° C.; and

sealing said growth chamber within graphite.